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ANCHOR QEA

October 18, 2022

Eric Winiecki
Project Coordinator
U.S. Environmental Protection Agency, Region 10
1200 Sixth Avenue, Suite 900
Seattle, Washington 98101

Re: Yakima Valley Dairies – Docket No. SWDA-10-2013-0080
Liberty/Bosma –Addendum for Soil Sampling at Bosma Lagoons 1, 2 and 3

Dear Eric,

This letter is provided on behalf of Liberty Dairy, LLC, and its associated dairy facility H&S Bosma Dairy (collectively, the Dairies). It provides the information requested in EPA Letter #288. Specifically this letter represents an addendum (Addendum) to the H&S Bosma Dairy Lagoon Nos. 1, 2, and 3 Soil Sampling Plan (SSP) as modified and approved by EPA on May 12, 2022.

1. Background

Lagoon Nos. 1, 2, and 3 at the H&S Bosma Dairy have historically served as animal waste storage ponds that contained manure, runoff from land areas contaminated with animal waste, and waste liquids from manure processing and other process operations. Between 2016 and 2021 the Dairy has undertaken a program to optimize its lagoon network. Lagoons 1, 2 and 3 are no longer required for manure or stormwater management and are therefore proposed by the Dairy for abandonment.

Prior to defining acceptable lagoon abandonment methods, EPA's modification to the SSP required that soil sampling data be collected beneath the three lagoons to document the distribution of subsurface soils containing elevated nitrogen, including both inorganic nitrogen (i.e., ammonia or nitrate) and organic nitrogen as determined from analysis of total kjeldahl nitrogen (TKN).

The SSP defines the locations within each lagoon from which samples are to be collected, either by backhoe test pit (shallow soil samples) or soil boring (deeper samples). Collected soil samples are to be analyzed for ammonia, nitrate and TKN at specific locations beneath the former lagoon bottom. Sampling must continue vertically at 1-foot intervals until either the target nitrogen concentration has been met in 4 consecutive samples or the groundwater table is encountered. The EPA-established target nitrogen concentration for this testing is 45 mg N/kg, measured as the sum of ammonia-N, nitrate-N and organic-N.

The SSP assumed that chemical analysis would be performed using the same laboratory that had been used for nutrient testing of soils and manure on the project, SoilTest Farm Consultants, Inc.

(SoilTest), a State of Washington-certified analytical laboratory and a North American Proficiency Testing-accredited laboratory located at 2925 Driggs Drive, Moses Lake, Washington.

The SSP established the required analysis methods as the following, with reference to the Dairy Facility Application Field Management Plan:

- **Ammonia (as nitrogen) by WCC S-3.50** (equivalent to United States Environmental Protection Agency (USEPA) method 350.1)
- **Nitrate (as nitrogen) by WCC S-3.10** (equivalent to EPA method 353.2)
- **TKN by WCC S-8.10** (equivalent to ASTM method ASTM D1426-15B modified)

In order to complete the SSP objectives, the nutrient data must accurately define inorganic and organic nitrogen levels at concentrations below the target concentration of 45 mg N/kg, considering all three analytes (ammonia, nitrate and organic nitrogen). If detection limits are elevated, or if the results are not accurate, then it will not be clear whether the target concentration has been met in a given soil sample.

2. Problem Definition

The problem necessitating this Addendum is the failure of the identified laboratory (SoilTest) to complete the required TKN analyses within project-required parameters. The solution to this problem as defined in Section 3 is to utilize a different laboratory to complete the required analyses within project-required parameters.

During the initial rounds of soil testing, Anchor QEA retained SoilTest to conduct analysis of the soil samples by the ammonia, nitrate and TKN test methods. During initial testing, the following problems were encountered and reported to EPA:

- **Failure to perform the requested TKN analysis:** Laboratory samples submitted for TKN analysis were not analyzed by the laboratory using the requested TKN test method. Instead the lab unilaterally substituted a different chemical analysis using the total nitrogen method. The lab did not consult with Anchor QEA prior to making that change.
- **Inability to reach the required method detection and reporting limits:** Given the EPA-specified target concentration of 45 mg N/kg, a TKN reporting limit equal to or less than 40 mg N/kg is required, with a desired detection limit lower than that value. SoilTest notified Anchor QEA that the best TKN performance that they can provide is a reporting limit of 200 mg N/kg and a detection limit of 50 mg N/kg. These values are insufficient to meet project requirements.

- **Limited capacity to perform TKN analysis:** SoilTest notified us that their throughput for TKN analysis is limited to 12 samples per day, including QA/QC samples. The lab stated that completion of the required work would take between 10 and 12 weeks to complete. This schedule is not acceptable, given EPA's emphasis of the need to complete the work as soon as practicable.

SoilTest initially proposed substitution of the total nitrogen test method in place of the SSP-required TKN analysis. However, the laboratory's indicated detection and reporting limits (80 mg N/kg and 400 mg N/kg respectively) are not sufficient to meet project requirements as defined in the SSP. Additionally, the total nitrogen testing results provided by the lab included significant apparent false positives as detected in field blanks submitted by Anchor QEA to the lab for testing. These field blanks consisted of laboratory-grade sand. No false positives were noted in the field blanks analyzed by SoilTest using the TKN test method; all four field blanks analyzed by the TKN test method yielded non-detect results at an indicated method detection limit of 50 mg N/kg. In contrast, the same blanks as analyzed by SoilTest using the total nitrogen test method yielded apparent false positive results at concentrations between 103 and 235 mg N/kg.

3. Resolution of the Problem

The proposed resolution of the problem is straight-forward. Anchor QEA proposes to substitute a second State-certified laboratory for the first. ALS Environmental (ALS) in Kelso, Washington is an NELAP and State of Washington-certified analytical laboratory. ALS routinely performs the TKN analysis, as well as ammonia and nitrate analysis on soils. ALS reports their standard TKN method detection and reporting limits as 8 mg N/kg and 40 mg N/kg, respectively. These values are well within project requirements as noted in Section 1 above. The laboratory also has a much higher sample throughput than SoilTest. ALS has estimated that the analyses required to complete SSP requirements can be completed by the end of October.

In order to provide a complete, comparable data set we propose to reanalyze all soil samples collected following issuance of the SSP for analysis of TKN. In two cases where archived samples are no longer available at SoilTest (i.e., 2 soil borings at Lagoon 3) we have recollected the soil samples (i.e., by repeating the two Lagoon 3 soil borings).

For newly-collected soil samples that have not previously been analyzed for ammonia and nitrate, we also propose to complete the ammonia and nitrate testing at ALS. The method detection and reporting limits for these parameters are similar between the two laboratories and are sufficient for meeting project requirements. Analyzing the samples at ALS avoids delays and chain-of-custody complications associated with sample splitting between the two labs.

Copies of the Washington State laboratory certifications and the ALS standard operating procedures for completion TKN analysis, ammonia and nitrate are provided in Attachment A.

4. Data Analysis and Reporting

All data for ammonia, nitrate and TKN analysis will be validated and reported to EPA. The proposed corrective action will result in some duplication of TKN testing results between the two labs. Where TKN results are available from prior SoilTest analyses, these will be validated and reported along-side the new data from ALS. Where "old" and "new" data differ, these differences will be called out in the report narrative and discussed.

Reporting requirements will remain as described in the SSP. The testing data for each lagoon will be submitted to EPA within 10 days of receipt of testing results for that lagoon.

Thank you for consideration of this Addendum.

Please do not hesitate to contact me if you have any questions or concerns. It has been a pleasure working with you on this project.

Sincerely,

Mark Larsen
Anchor QEA, LLC

cc:

Don Clabaugh, U.S. Environmental Protection Agency
Donald Brown, U.S. Environmental Protection Agency
Jennifer MacDonald, U.S. Environmental Protection Agency
John (Mathew) Moore, U.S. Environmental Protection Agency
Ed Kowalski, U.S. Environmental Protection Agency
Bill Dunbar, U.S. Environmental Protection Agency
Lucy Edmonson, U.S. Environmental Protection Agency
Kristi Geris, Anchor QEA, LLC
Josh Sexton, Anchor QEA, LLC
Henry Bosma, Liberty Dairy, LLC
Meredith Weinberg, Perkins Coie, LLP

Attachments

Attachment A ALS Lab Certifications and SOPs

The State of Washington Department of Ecology

ALS Environmental - Kelso

Kelso, WA

has complied with provisions set forth in Chapter 173-50 WAC and is hereby recognized by the Department of Ecology as an ACCREDITED LABORATORY for the analytical parameters listed on the accompanying Scope of Accreditation.

This certificate is effective July 9, 2022 and shall expire July 8, 2023.

Witnessed under my hand on July 20, 2022.

Rebecca Wood
Lab Accreditation Unit Supervisor

Laboratory ID
C544

WASHINGTON STATE DEPARTMENT OF ECOLOGY

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

SCOPE OF ACCREDITATION

ALS Environmental - Kelso

Kelso, WA

is accredited for the analytes listed below using the methods indicated. Full accreditation is granted unless stated otherwise in a note. EPA is the U.S. Environmental Protection Agency. SM is "Standard Methods for the Examination of Water and Wastewater." SM refers to EPA approved method versions. ASTM is the American Society for Testing and Materials. USGS is the U.S. Geological Survey. AOAC is the Association of Official Analytical Chemists. Other references are described in notes.

Matrix/Analyte	Method	Notes
Drinking Water		
Chloride	EPA 300.0_2.1_1993	4
Fluoride	EPA 300.0_2.1_1993	4
Nitrate	EPA 300.0_2.1_1993	4
Nitrite	EPA 300.0_2.1_1993	4
Sulfate	EPA 300.0_2.1_1993	4
Cyanide, Total	EPA 335.4_1_1993	4,5
Nitrate	EPA 353.2_2_1993	4
Nitrite	EPA 353.2_2_1993	4
Color	SM 2120 B-2011	4
Alkalinity	SM 2320 B-2011	4
Specific Conductance	SM 2510 B-2011	4
Solids, Total Dissolved	SM 2540 C-2011	4
Cyanide, Total	SM 4500-CN^- E-2011	
Fluoride	SM 4500-F^- C-2011	4
pH	SM 4500-H+ B-2011	1,4
Orthophosphate	SM 4500-P E-2011	4
Total Organic Carbon	SM 5310 C-2011	4
Aluminum	EPA 200.7_4.4_1994	4
Barium	EPA 200.7_4.4_1994	4
Beryllium	EPA 200.7_4.4_1994	4
Boron	EPA 200.7_4.4_1994	4
Cadmium	EPA 200.7_4.4_1994	4
Calcium	EPA 200.7_4.4_1994	4
Chromium	EPA 200.7_4.4_1994	4
Copper	EPA 200.7_4.4_1994	4
Iron	EPA 200.7_4.4_1994	4

Matrix/Analyte	Method	Notes
Drinking Water		
Magnesium	EPA 200.7_4.4_1994	4
Manganese	EPA 200.7_4.4_1994	4
Molybdenum	EPA 200.7_4.4_1994	4
Nickel	EPA 200.7_4.4_1994	4
Potassium	EPA 200.7_4.4_1994	4
Silica	EPA 200.7_4.4_1994	4
Silver	EPA 200.7_4.4_1994	4
Sodium	EPA 200.7_4.4_1994	4
Vanadium	EPA 200.7_4.4_1994	4
Zinc	EPA 200.7_4.4_1994	4
Aluminum	EPA 200.8_5.4_1994	4
Antimony	EPA 200.8_5.4_1994	4
Arsenic	EPA 200.8_5.4_1994	4
Barium	EPA 200.8_5.4_1994	4
Beryllium	EPA 200.8_5.4_1994	4
Cadmium	EPA 200.8_5.4_1994	4
Chromium	EPA 200.8_5.4_1994	4
Copper	EPA 200.8_5.4_1994	4
Lead	EPA 200.8_5.4_1994	4
Manganese	EPA 200.8_5.4_1994	4
Nickel	EPA 200.8_5.4_1994	4
Selenium	EPA 200.8_5.4_1994	4
Silver	EPA 200.8_5.4_1994	4
Thallium	EPA 200.8_5.4_1994	4
Uranium	EPA 200.8_5.4_1994	
Zinc	EPA 200.8_5.4_1994	
Mercury	EPA 245.1_3_1994	4
Hardness, Total (as CaCO3)	SM 2340 B-2011	4
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1_1.1_1995	4
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1_1.1_1995	4
Bromoacetic acid (MBAA, BAA)	EPA 552.2_1_1995	4
Bromochloroacetic acid (BCAA)	EPA 552.2_1_1995	4
Chloroacetic acid (MCAA, CAA)	EPA 552.2_1_1995	4
Dibromoacetic acid (DBAA)	EPA 552.2_1_1995	4
Dichloroacetic acid (DCAA)	EPA 552.2_1_1995	4
Total haloacetic acids (HAA5)	EPA 552.2_1_1995	4
Trichloroacetic acid (TCAA)	EPA 552.2_1_1995	4

Matrix/Analyte	Method	Notes
Drinking Water		
1,1,1,2-Tetrachloroethane	EPA 524.2_4.1_1995	4
1,1,1-Trichloroethane	EPA 524.2_4.1_1995	4
1,1,2,2-Tetrachloroethane	EPA 524.2_4.1_1995	4
1,1,2-Trichloroethane	EPA 524.2_4.1_1995	4
1,1-Dichloroethane	EPA 524.2_4.1_1995	4
1,1-Dichloroethylene	EPA 524.2_4.1_1995	4
1,1-Dichloropropene	EPA 524.2_4.1_1995	4
1,2,3-Trichlorobenzene	EPA 524.2_4.1_1995	4
1,2,3-Trichloropropane	EPA 524.2_4.1_1995	4
1,2,4-Trichlorobenzene	EPA 524.2_4.1_1995	4
1,2,4-Trimethylbenzene	EPA 524.2_4.1_1995	4
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 524.2_4.1_1995	14
1,2-Dichlorobenzene	EPA 524.2_4.1_1995	4
1,2-Dichloroethane (Ethylene dichloride)	EPA 524.2_4.1_1995	4
1,2-Dichloropropane	EPA 524.2_4.1_1995	4
1,3,5-Trimethylbenzene	EPA 524.2_4.1_1995	4
1,3-Dichlorobenzene	EPA 524.2_4.1_1995	4
1,3-Dichloropropane	EPA 524.2_4.1_1995	4
1,4-Dichlorobenzene	EPA 524.2_4.1_1995	4
2,2-Dichloropropane	EPA 524.2_4.1_1995	4
2-Butanone (Methyl ethyl ketone, MEK)	EPA 524.2_4.1_1995	11
2-Chlorotoluene	EPA 524.2_4.1_1995	4
2-Hexanone	EPA 524.2_4.1_1995	11
4-Chlorotoluene	EPA 524.2_4.1_1995	4
4-Isopropyltoluene (p-Cymene)	EPA 524.2_4.1_1995	4
4-Methyl-2-pentanone (MIBK)	EPA 524.2_4.1_1995	11
Acetone	EPA 524.2_4.1_1995	11
Benzene	EPA 524.2_4.1_1995	4
Bromobenzene	EPA 524.2_4.1_1995	4
Bromochloromethane	EPA 524.2_4.1_1995	4
Bromodichloromethane	EPA 524.2_4.1_1995	4
Bromoform	EPA 524.2_4.1_1995	4
Carbon disulfide	EPA 524.2_4.1_1995	11
Carbon tetrachloride	EPA 524.2_4.1_1995	4
Chlorobenzene	EPA 524.2_4.1_1995	4
Chlorodibromomethane	EPA 524.2_4.1_1995	4
Chloroethane (Ethyl chloride)	EPA 524.2_4.1_1995	4

Matrix/Analyte	Method	Notes
Drinking Water		
Chloroform	EPA 524.2_4.1_1995	4
cis-1,2-Dichloroethylene	EPA 524.2_4.1_1995	4
cis-1,3-Dichloropropene	EPA 524.2_4.1_1995	4
Dibromomethane	EPA 524.2_4.1_1995	4
Dichlorodifluoromethane (Freon-12)	EPA 524.2_4.1_1995	4
Dichloromethane (DCM, Methylene chloride)	EPA 524.2_4.1_1995	4
Ethylbenzene	EPA 524.2_4.1_1995	4
Hexachlorobutadiene	EPA 524.2_4.1_1995	4
Isopropylbenzene	EPA 524.2_4.1_1995	4
m+p-xylene	EPA 524.2_4.1_1995	4
Methyl bromide (Bromomethane)	EPA 524.2_4.1_1995	4
Methyl chloride (Chloromethane)	EPA 524.2_4.1_1995	4
Methyl tert-butyl ether (MTBE)	EPA 524.2_4.1_1995	4
Naphthalene	EPA 524.2_4.1_1995	4
n-Butylbenzene	EPA 524.2_4.1_1995	4
n-Propylbenzene	EPA 524.2_4.1_1995	4
o-Xylene	EPA 524.2_4.1_1995	4
sec-Butylbenzene	EPA 524.2_4.1_1995	4
Styrene	EPA 524.2_4.1_1995	4
tert-Butylbenzene	EPA 524.2_4.1_1995	4
Tetrachloroethylene (Perchloroethylene)	EPA 524.2_4.1_1995	4
Toluene	EPA 524.2_4.1_1995	4
Total Trihalomethanes	EPA 524.2_4.1_1995	4
trans-1,2-Dichloroethylene	EPA 524.2_4.1_1995	4
trans-1,3-Dichloropropylene	EPA 524.2_4.1_1995	4
Trichloroethene (Trichloroethylene)	EPA 524.2_4.1_1995	4
Trichlorofluoromethane (Freon 11)	EPA 524.2_4.1_1995	4
Vinyl chloride	EPA 524.2_4.1_1995	4
Xylene (total)	EPA 524.2_4.1_1995	4
N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA)	EPA 537_1.1_2009	3,4
N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA)	EPA 537_1.1_2009	3,4
Perfluorobutane sulfonic acid (PFBS)	EPA 537_1.1_2009	3,4
Perfluorodecanoic acid (PFDA)	EPA 537_1.1_2009	3,4
Perfluorododecanoic acid (PFDoA)	EPA 537_1.1_2009	3,4
Perfluoroheptanoic acid (PFHpA)	EPA 537_1.1_2009	3,4
Perfluorohexane sulfonic acid (PFHxS)	EPA 537_1.1_2009	3,4
Perfluorohexanoic acid (PFHxA)	EPA 537_1.1_2009	3,4

Matrix/Analyte	Method	Notes
Drinking Water		
Perfluorononanoic acid (PFNA)	EPA 537_1.1_2009	3,4
Perfluorooctane sulfonic acid (PFOS)	EPA 537_1.1_2009	3,4
Perfluorooctanoic acid (PFOA)	EPA 537_1.1_2009	3,4
Perfluorotetradecanoic acid (PFTeDA)	EPA 537_1.1_2009	3,4
Perfluorotridecanoic acid (PFTrDA)	EPA 537_1.1_2009	3,4
Perfluoroundecanoic acid (PFUnA)	EPA 537_1.1_2009	3,4
Heterotrophic Bacteria	SM 9215 B (PCA)	4
Fecal coliform-count	SM 9222 D (mFC)-06	
Total coli/E.coli - detect	SM 9223 B Colilert 18® (PA)	4
E.coli-count	SM 9223 B Colilert 18® QTray®	4
Total coli/E.coli - detect	SM 9223 B Colilert® 24 (PA)	4
E.coli-count	SM 9223 B Colilert® 24 QTray®	4
Non-Potable Water		
Solids, Total Volatile	EPA 160.4_1971	4
Adsorbable Organic Halides (AOX)	EPA 1650C_1997	4
n-Hexane Extractable Material (O&G)	EPA 1664A (SGT-HEM)	4
n-Hexane Extractable Material (O&G)	EPA 1664A_1_1999	4
Turbidity	EPA 180.1_2_1993	4
Bromide	EPA 300.0_2.1_1993	4,5
Chloride	EPA 300.0_2.1_1993	4
Fluoride	EPA 300.0_2.1_1993	4
Nitrate	EPA 300.0_2.1_1993	4
Nitrite	EPA 300.0_2.1_1993	4
Sulfate	EPA 300.0_2.1_1993	4
Cyanide, Total	EPA 335.4_1_1993	4
Ammonia	EPA 350.1_2_1993	
Nitrate	EPA 353.2_2_1993	4
Nitrate	EPA 353.2_2_1993	4
Nitrate + Nitrite	EPA 353.2_2_1993	4
Nitrate + Nitrite	EPA 353.2_2_1993	4
Nitrite	EPA 353.2_2_1993	4
Orthophosphate	EPA 365.3_1978	4
Phosphorus, total	EPA 365.3_1978	4
Phenolics, Total	EPA 420.1_1978	4
Formaldehyde	NCASI 98.01	
Chlorophyll a	SM 10200H-2011	
Color	SM 2120 B-2011	4

Matrix/Analyte	Method	Notes
Non-Potable Water		
Acidity	SM 2310 B-2011	4
Alkalinity	SM 2320 B-2011	4
Hardness (calc.)	SM 2340 B-2011	4
Hardness, Total (as CaCO3)	SM 2340 C-2011	4
Specific Conductance	SM 2510 B-2011	4
Solids, Total	SM 2540 B-2011	4
Solids, Total Dissolved	SM 2540 C-2011	4
Solids, Total Suspended	SM 2540 D-2011	4
Solids, Settleable	SM 2540 F-2011	4
Chromium, Hexavalent	SM 3500-Cr B-2011	
Chloride	SM 4500-Cl^- C-2011	4
Cyanide, Total	SM 4500-CN^- E-2011	4
Fluoride	SM 4500-F^- C-2011	4
pH	SM 4500-H+ B-2011	1,4
Ammonia	SM 4500-NH3 E-2011	4
Ammonia	SM 4500-NH3 G-2011	4
Sulfide	SM 4500-$S2^-$ D-2011	4
Sulfide	SM 4500-$S2^-$ F-2011	4
Biochemical Oxygen Demand (BOD)	SM 5210 B-2011	4
Chemical Oxygen Demand (COD)	SM 5220 C-2011	4
Total Organic Carbon	SM 5310 C-2011	4
Anionic Surfactants (MBAS)	SM 5540 C-2011	4
Tannin & Lignin	SM 5550 B-93	4
Methyl Mercury	EPA 1630	4
Mercury	EPA 1631 E-02	4
Arsenic	EPA 1632A 1998	4
Arsenic (III)	EPA 1632A 1998	4
Arsenic (V)	EPA 1632A 1998	
Aluminum	EPA 200.7_4.4_1994	4
Antimony	EPA 200.7_4.4_1994	4
Arsenic	EPA 200.7_4.4_1994	4
Barium	EPA 200.7_4.4_1994	4
Beryllium	EPA 200.7_4.4_1994	4
Boron	EPA 200.7_4.4_1994	4
Cadmium	EPA 200.7_4.4_1994	4
Calcium	EPA 200.7_4.4_1994	4
Chromium	EPA 200.7_4.4_1994	4

Matrix/Analyte	Method	Notes
Non-Potable Water		
Cobalt	EPA 200.7_4.4_1994	4
Copper	EPA 200.7_4.4_1994	4
Hardness, Total (as CaCO3)	EPA 200.7_4.4_1994	4
Iron	EPA 200.7_4.4_1994	4
Lead	EPA 200.7_4.4_1994	4
Magnesium	EPA 200.7_4.4_1994	4
Manganese	EPA 200.7_4.4_1994	4
Molybdenum	EPA 200.7_4.4_1994	4
Nickel	EPA 200.7_4.4_1994	4
Potassium	EPA 200.7_4.4_1994	4
Selenium	EPA 200.7_4.4_1994	4
Silica	EPA 200.7_4.4_1994	4
Silver	EPA 200.7_4.4_1994	4
Sodium	EPA 200.7_4.4_1994	4
Strontium	EPA 200.7_4.4_1994	4
Thallium	EPA 200.7_4.4_1994	
Tin	EPA 200.7_4.4_1994	4
Titanium	EPA 200.7_4.4_1994	4
Vanadium	EPA 200.7_4.4_1994	4
Zinc	EPA 200.7_4.4_1994	4
Aluminum	EPA 200.8_5.4_1994	4
Antimony	EPA 200.8_5.4_1994	4
Arsenic	EPA 200.8_5.4_1994	4
Barium	EPA 200.8_5.4_1994	4
Beryllium	EPA 200.8_5.4_1994	4
Cadmium	EPA 200.8_5.4_1994	4
Chromium	EPA 200.8_5.4_1994	4
Cobalt	EPA 200.8_5.4_1994	4
Copper	EPA 200.8_5.4_1994	4
Iron	EPA 200.8_5.4_1994	4
Lead	EPA 200.8_5.4_1994	4
Manganese	EPA 200.8_5.4_1994	4
Molybdenum	EPA 200.8_5.4_1994	4
Nickel	EPA 200.8_5.4_1994	4
Selenium	EPA 200.8_5.4_1994	4
Silver	EPA 200.8_5.4_1994	4
Thallium	EPA 200.8_5.4_1994	4

Matrix/Analyte	Method	Notes
Non-Potable Water		
Tin	EPA 200.8_5.4_1994	
Vanadium	EPA 200.8_5.4_1994	4
Zinc	EPA 200.8_5.4_1994	4
Mercury	EPA 245.1_3_1994	4
4,4'-DDD	EPA 608.3	4,8
4,4'-DDE	EPA 608.3	4,8
4,4'-DDT	EPA 608.3	4,8
Aldrin	EPA 608.3	4,8
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608.3	4,8
alpha-Chlordane	EPA 608.3	4,8
Aroclor-1016 (PCB-1016)	EPA 608.3	4,8
Aroclor-1221 (PCB-1221)	EPA 608.3	4,8
Aroclor-1232 (PCB-1232)	EPA 608.3	4,8
Aroclor-1242 (PCB-1242)	EPA 608.3	4,8
Aroclor-1248 (PCB-1248)	EPA 608.3	4,8
Aroclor-1254 (PCB-1254)	EPA 608.3	4,8
Aroclor-1260 (PCB-1260)	EPA 608.3	4,8
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608.3	4,8
Chlordane (tech.)	EPA 608.3	4,8
delta-BHC	EPA 608.3	4,8
Dieldrin	EPA 608.3	4,8
Endosulfan I	EPA 608.3	4,8
Endosulfan II	EPA 608.3	4,8
Endosulfan sulfate	EPA 608.3	4,8
Endrin	EPA 608.3	4,8
Endrin aldehyde	EPA 608.3	4,8
Endrin ketone	EPA 608.3	4,8
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608.3	4,8
gamma-Chlordane	EPA 608.3	4,8
Heptachlor	EPA 608.3	4,8
Heptachlor epoxide	EPA 608.3	4,8
Methoxychlor	EPA 608.3	4,8
Toxaphene (Chlorinated camphene)	EPA 608.3	4,8
Organo-tins	Krone 1988	
Methanol	NCASI 94.03	
2-Butanone (Methyl ethyl ketone, MEK)	NCASI DI/HAPS-99.01	
Acetaldehyde	NCASI DI/HAPS-99.01	

Matrix/Analyte	Method	Notes
Non-Potable Water		
Methanol	NCASI DI/HAPS-99.01	
Propionaldehyde	NCASI DI/HAPS-99.01	
1H,1H,2H,2H,-Perfluorodecanesulfonic acid (8:2 FTS)	ALS LCP-PFC Rev. 11.0	4
1H,1H,2H,2H,-Perfluorooctansulfonic acid (6:2 FTS)	ALS LCP-PFC Rev. 11.0	4
1H,1H,2H,2H-Perfluorododecane sulfonic acid (10:2-FTS)	ALS LCP-PFC Rev. 11.0	4
1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 FTS)	ALS LCP-PFC Rev. 11.0	4
Hexafluoropropylene oxide dimer acid (HFPO-DA)	ALS LCP-PFC Rev. 11.0	4
N-Ethylperfluorooctane sulfonamide (EtFOSA)	ALS LCP-PFC Rev. 11.0	4
N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA)	ALS LCP-PFC Rev. 11.0	4
N-Ethylperfluorooctanesulfonamidoethanol (EtFOSE)	ALS LCP-PFC Rev. 11.0	4
N-Methylperfluorooctane sulfonamide (MeFOSA)	ALS LCP-PFC Rev. 11.0	4
N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA)	ALS LCP-PFC Rev. 11.0	4
N-Methylperfluorooctanesulfonamido ethanol (MeFOSE)	ALS LCP-PFC Rev. 11.0	4
Perfluorobutane sulfonic acid (PFBS)	ALS LCP-PFC Rev. 11.0	4
Perfluorobutanoic acid (PFBA)	ALS LCP-PFC Rev. 11.0	4
Perfluorodecane sulfonic acid (PFDS)	ALS LCP-PFC Rev. 11.0	4
Perfluorodecanoic acid (PFDA)	ALS LCP-PFC Rev. 11.0	4
Perfluorododecanoic acid (PFDoA)	ALS LCP-PFC Rev. 11.0	4
Perfluoroheptane sulfonic acid (PFHpS)	ALS LCP-PFC Rev. 11.0	4
Perfluoroheptanoic acid (PFHpA)	ALS LCP-PFC Rev. 11.0	4
Perfluorohexane sulfonic acid (PFHxS)	ALS LCP-PFC Rev. 11.0	4
Perfluorohexanoic acid (PFHxA)	ALS LCP-PFC Rev. 11.0	4
Perfluorononanoic acid (PFNA)	ALS LCP-PFC Rev. 11.0	4
Perfluorooctane sulfonamide (PFOSA)	ALS LCP-PFC Rev. 11.0	4
Perfluorooctane sulfonic acid (PFOS)	ALS LCP-PFC Rev. 11.0	4
Perfluorooctanoic acid (PFOA)	ALS LCP-PFC Rev. 11.0	4
Perfluoropentanoic acid (PFPeA)	ALS LCP-PFC Rev. 11.0	4
Perfluorotetradecanoic acid (PFTeDA)	ALS LCP-PFC Rev. 11.0	4
Perfluorotridecanoic acid (PFTrDA)	ALS LCP-PFC Rev. 11.0	4
Perfluoroundecanoic acid (PFUnA)	ALS LCP-PFC Rev. 11.0	4
2,3,4,6-Tetrachlorophenol	EPA 1653A_1997	4
2,4,5-Trichlorophenol	EPA 1653A_1997	4
2,4,6-Trichlorophenol	EPA 1653A_1997	4
3,4,5-Trichlorocatechol	EPA 1653A_1997	4
3,4,5-Trichloroguaiacol	EPA 1653A_1997	4
3,4,6-Trichlorocatechol	EPA 1653A_1997	4
3,4,6-Trichloroguaiacol	EPA 1653A_1997	4

Matrix/Analyte	Method	Notes
Non-Potable Water		
4,5,6-Trichloroguaiacol	EPA 1653A_1997	4
Pentachlorophenol	EPA 1653A_1997	4
Tetrachlorocatechol	EPA 1653A_1997	4
Tetrachloroguaiacol	EPA 1653A_1997	4
Trichlorosyringol	EPA 1653A_1997	4
Acetaminophen	EPA 1694_2007	4
Caffeine	EPA 1694_2007	4
Carbamazepine	EPA 1694_2007	4
Fluoxetine	EPA 1694_2007	4
Gemfibrozil	EPA 1694_2007	4
Ibuprofen	EPA 1694_2007	4
Naproxen	EPA 1694_2007	4
Sulfamethoxazole	EPA 1694_2007	4
Triclosan	EPA 1694_2007	4
Trimethoprim	EPA 1694_2007	4
1,1,1,2-Tetrachloroethane	EPA 624.1	9
1,1,1-Trichloro-2,2,2-trifluoroethane	EPA 624.1	4,9
1,1,1-Trichloroethane	EPA 624.1	9
1,1,2,2-Tetrachloroethane	EPA 624.1	4,9
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA 624.1	4,9
1,1,2-Trichloroethane	EPA 624.1	4,9
1,1-Dichloroethane	EPA 624.1	4,9
1,1-Dichloroethylene	EPA 624.1	4,9
1,1-Dichloropropene	EPA 624.1	4,9
1,2,3-Trichlorobenzene	EPA 624.1	4,9
1,2,3-Trichloropropane	EPA 624.1	4,9
1,2,4-Trimethylbenzene	EPA 624.1	4,9
1,2-Dibromo-3-chloropropane (DBCP)	EPA 624.1	4,9
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 624.1	4,9
1,2-Dichlorobenzene	EPA 624.1	4,9
1,2-Dichloroethane (Ethylene dichloride)	EPA 624.1	4,9
1,2-Dichloropropane	EPA 624.1	4,9
1,3,5-Trimethylbenzene	EPA 624.1	4,9
1,3-Dichlorobenzene	EPA 624.1	4,9
1,3-Dichloropropane	EPA 624.1	4,9
1,4-Dichlorobenzene	EPA 624.1	4,9
2,2-Dichloropropane	EPA 624.1	4,9

Matrix/Analyte	Method	Notes
Non-Potable Water		
2-Butanone (Methyl ethyl ketone, MEK)	EPA 624.1	4,9
2-Chloroethyl vinyl ether	EPA 624.1	4,9
2-Chlorotoluene	EPA 624.1	4,9
2-Hexanone	EPA 624.1	4,9
4-Chlorotoluene	EPA 624.1	4,9
4-Methyl-2-pentanone (MIBK)	EPA 624.1	4,9
Acetone	EPA 624.1	4,9
Acetonitrile	EPA 624.1	4,9
Acrolein (Propenal)	EPA 624.1	4,9
Acrylonitrile	EPA 624.1	4,9
Benzene	EPA 624.1	4,9
Bromobenzene	EPA 624.1	4,9
Bromochloromethane	EPA 624.1	4,9
Bromodichloromethane	EPA 624.1	4,9
Bromoform	EPA 624.1	3,9
Carbon disulfide	EPA 624.1	4,9
Carbon tetrachloride	EPA 624.1	4,9
Chlorobenzene	EPA 624.1	4,9
Chlorodibromomethane	EPA 624.1	4,9
Chloroethane (Ethyl chloride)	EPA 624.1	4,9
Chloroform	EPA 624.1	4,9
cis-1,2-Dichloroethylene	EPA 624.1	4,9
cis-1,3-Dichloropropene	EPA 624.1	4,9
Dibromomethane	EPA 624.1	4,9
Dichlorofluoromethane (Freon 21)	EPA 624.1	4,9
Dichloromethane (DCM, Methylene chloride)	EPA 624.1	4,9
Diethyl ether	EPA 624.1	4,9
Ethylbenzene	EPA 624.1	4,9
Methyl bromide (Bromomethane)	EPA 624.1	9
Methyl chloride (Chloromethane)	EPA 624.1	4,9
Methyl tert-butyl ether (MTBE)	EPA 624.1	4,9
Methylene chloride (Dichloromethane)	EPA 624.1	4,9
n-Butylbenzene	EPA 624.1	4,9
n-Propylbenzene	EPA 624.1	4,9
o-Xylene	EPA 624.1	4,9
sec-Butylbenzene	EPA 624.1	4,9
Styrene	EPA 624.1	4,9

Matrix/Analyte	Method	Notes
Non-Potable Water		
Tetrachloroethylene (Perchloroethylene)	EPA 624.1	4,9
Toluene	EPA 624.1	4,9
trans-1,2-Dichloroethylene	EPA 624.1	4,9
trans-1,3-Dichloropropylene	EPA 624.1	4,9
Trichloroethene (Trichloroethylene)	EPA 624.1	4,9
Trichlorofluoromethane (Freon 11)	EPA 624.1	4,9
Vinyl acetate	EPA 624.1	4,9
Vinyl chloride	EPA 624.1	4,9
1,2,4-Trichlorobenzene	EPA 625.1	4,10
1,2-Diphenylhydrazine	EPA 625.1	4,10
2,4,6-Trichlorophenol	EPA 625.1	4,10
2,4-Dichlorophenol	EPA 625.1	4,10
2,4-Dimethylphenol	EPA 625.1	4,10
2,4-Dinitrophenol	EPA 625.1	4,10
2,4-Dinitrotoluene (2,4-DNT)	EPA 625.1	4,10
2,6-Dinitrotoluene (2,6-DNT)	EPA 625.1	4,10
2-Chloronaphthalene	EPA 625.1	4,10
2-Chlorophenol	EPA 625.1	4,10
2-Nitrophenol	EPA 625.1	4,10
3,3'-Dichlorobenzidine	EPA 625.1	4,10
4,6-Dinitro-2-methylphenol	EPA 625.1	10
4-Bromophenyl phenyl ether (BDE-3)	EPA 625.1	4,10
4-Chloro-3-methylphenol	EPA 625.1	4,10
4-Chlorophenyl phenylether	EPA 625.1	4,10
4-Nitrophenol	EPA 625.1	4,10
Acenaphthene	EPA 625.1	4,10
Acenaphthylene	EPA 625.1	4,10
Anthracene	EPA 625.1	4,10
Atrazine	EPA 625.1	10
Benzidine	EPA 625.1	4,10
Benzo(a)anthracene	EPA 625.1	4,10
Benzo(a)pyrene	EPA 625.1	4,10
Benzo(g,h,i)perylene	EPA 625.1	4,10
Benzo(k)fluoranthene	EPA 625.1	4,10
Benzo[b]fluoranthene	EPA 625.1	4,10
Benzoic acid	EPA 625.1	4,10
Biphenyl	EPA 625.1	10

Matrix/Analyte	Method	Notes
Non-Potable Water		
bis(2-Chloroethoxy)methane	EPA 625.1	4,10
bis(2-Chloroethyl) ether	EPA 625.1	4,10
bis(2-Chloroisopropyl) ether	EPA 625.1	10
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625.1	10
Butyl benzyl phthalate	EPA 625.1	4,10
Carbazole	EPA 625.1	4,10
Chrysene	EPA 625.1	4,10
Dibenz(a,h) anthracene	EPA 625.1	4,10
Dibenzofuran	EPA 625.1	4,10
Diethyl phthalate	EPA 625.1	4,10
Dimethyl phthalate	EPA 625.1	4,10
Di-n-butyl phthalate	EPA 625.1	4,10
Di-n-octyl phthalate	EPA 625.1	4,10
Fluoranthene	EPA 625.1	4,10
Fluorene	EPA 625.1	4,10
Hexachlorobenzene	EPA 625.1	4,10
Hexachlorobutadiene	EPA 625.1	4,10
Hexachlorocyclopentadiene	EPA 625.1	4,10
Hexachloroethane	EPA 625.1	4,10
Indeno(1,2,3-cd) pyrene	EPA 625.1	4,10
Isophorone	EPA 625.1	4,10
Naphthalene	EPA 625.1	4,10
Nitrobenzene	EPA 625.1	4,10
N-Nitrosodimethylamine	EPA 625.1	4,10
N-Nitroso-di-n-propylamine	EPA 625.1	4,10
N-Nitrosodiphenylamine	EPA 625.1	4,10
Pentachlorophenol	EPA 625.1	4,10
Phenanthrene	EPA 625.1	4,10
Phenol	EPA 625.1	4,10
Pyrene	EPA 625.1	4,10
Pyridine	EPA 625.1	4,10
Heterotrophic Bacteria	SM 9215 B (PCA)	4
Fecal coliform-count	SM 9221 B+E1+C (LTB/BGB/EC-MPN)	4
Total coliforms-count	SM 9221 B+E1+C (LTB/BGB/EC-MPN)	4
Fecal coliform-count	SM 9222 D (mFC)-06	4
E.coli-count	SM 9223 B Colilert 18® QTray®	4
E.coli-count	SM 9223 B Colilert® 24 QTray®	4

Matrix/Analyte	Method	Notes
Non-Potable Water		
Enterococci	SM 9230 D Enterolert®	
Solid and Chemical Materials		
Nitrogen, Total Kjeldahl	ASTM D3590-02	4
Total Organic Carbon	ASTM D4129-05	4
Solids, Total Volatile	EPA 160.4_1971	
Ammonia	EPA 350.1_2_1993	4
Nitrate	EPA 353.2_2_1993	4
Nitrite	EPA 353.2_2_1993	4
Orthophosphate	EPA 365.3_1978	4
Phosphorus, total	EPA 365.3_1978	4
Chromium, Hexavalent	EPA 7196A_1_1992	4
Cyanide, Total	EPA 9012 B-04	4
Sulfide	EPA 9030B_2_1996	4
pH	EPA 9045D_2002	4
Chloride	EPA 9056A_(02/07)	4
Fluoride	EPA 9056A_(02/07)	4
Nitrate	EPA 9056A_(02/07)	4
Sulfate	EPA 9056A_(02/07)	4
Total Organic Carbon	EPA 9060A_1_2004	
n-Hexane Extractable Material (O&G)	EPA 9071 A	4
Solids, Total	SM 2540 B-2011	
Solids, Total, Fixed and Volatile	SM 2540 G-2011	
Chemical Oxygen Demand (COD)	SM 5220 C-2011	2
Methyl Mercury	EPA 1630	4
Mercury	EPA 1631 E-02	4
Arsenic	EPA 1632A 1998	
Arsenic (III)	EPA 1632A 1998	
Arsenic (V)	EPA 1632A 1998	
Aluminum	EPA 200.7_4.4_1994	
Antimony	EPA 200.7_4.4_1994	
Arsenic	EPA 200.7_4.4_1994	
Barium	EPA 200.7_4.4_1994	
Beryllium	EPA 200.7_4.4_1994	
Boron	EPA 200.7_4.4_1994	
Cadmium	EPA 200.7_4.4_1994	
Calcium	EPA 200.7_4.4_1994	
Chromium	EPA 200.7_4.4_1994	

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
Cobalt	EPA 200.7_4.4_1994	
Copper	EPA 200.7_4.4_1994	
Iron	EPA 200.7_4.4_1994	
Lead	EPA 200.7_4.4_1994	
Magnesium	EPA 200.7_4.4_1994	
Manganese	EPA 200.7_4.4_1994	
Molybdenum	EPA 200.7_4.4_1994	
Nickel	EPA 200.7_4.4_1994	
Potassium	EPA 200.7_4.4_1994	
Selenium	EPA 200.7_4.4_1994	
Silica	EPA 200.7_4.4_1994	2
Silver	EPA 200.7_4.4_1994	
Sodium	EPA 200.7_4.4_1994	
Strontium	EPA 200.7_4.4_1994	
Thallium	EPA 200.7_4.4_1994	
Tin	EPA 200.7_4.4_1994	
Titanium	EPA 200.7_4.4_1994	
Vanadium	EPA 200.7_4.4_1994	
Zinc	EPA 200.7_4.4_1994	
Aluminum	EPA 200.8_5.4_1994	
Antimony	EPA 200.8_5.4_1994	
Arsenic	EPA 200.8_5.4_1994	
Barium	EPA 200.8_5.4_1994	
Beryllium	EPA 200.8_5.4_1994	
Cadmium	EPA 200.8_5.4_1994	
Chromium	EPA 200.8_5.4_1994	
Cobalt	EPA 200.8_5.4_1994	
Copper	EPA 200.8_5.4_1994	
Iron	EPA 200.8_5.4_1994	
Lead	EPA 200.8_5.4_1994	
Manganese	EPA 200.8_5.4_1994	
Molybdenum	EPA 200.8_5.4_1994	
Nickel	EPA 200.8_5.4_1994	
Selenium	EPA 200.8_5.4_1994	
Silver	EPA 200.8_5.4_1994	
Thallium	EPA 200.8_5.4_1994	
Uranium	EPA 200.8_5.4_1994	2

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
Vanadium	EPA 200.8_5.4_1994	
Zinc	EPA 200.8_5.4_1994	
Aluminum	EPA 6010D_(7/14)	4
Antimony	EPA 6010D_(7/14)	4
Arsenic	EPA 6010D_(7/14)	4
Barium	EPA 6010D_(7/14)	4
Beryllium	EPA 6010D_(7/14)	4
Boron	EPA 6010D_(7/14)	4
Cadmium	EPA 6010D_(7/14)	4
Calcium	EPA 6010D_(7/14)	4
Chromium	EPA 6010D_(7/14)	4
Cobalt	EPA 6010D_(7/14)	4
Copper	EPA 6010D_(7/14)	4
Iron	EPA 6010D_(7/14)	4
Lead	EPA 6010D_(7/14)	4
Magnesium	EPA 6010D_(7/14)	4
Manganese	EPA 6010D_(7/14)	4
Molybdenum	EPA 6010D_(7/14)	4
Nickel	EPA 6010D_(7/14)	4
Potassium	EPA 6010D_(7/14)	4
Selenium	EPA 6010D_(7/14)	4
Silver	EPA 6010D_(7/14)	4
Sodium	EPA 6010D_(7/14)	4
Strontium	EPA 6010D_(7/14)	4
Thallium	EPA 6010D_(7/14)	4
Vanadium	EPA 6010D_(7/14)	4
Zinc	EPA 6010D_(7/14)	4
Aluminum	EPA 6020B_(7/14)	4
Antimony	EPA 6020B_(7/14)	4
Arsenic	EPA 6020B_(7/14)	4
Barium	EPA 6020B_(7/14)	4
Beryllium	EPA 6020B_(7/14)	4
Cadmium	EPA 6020B_(7/14)	4
Chromium	EPA 6020B_(7/14)	4
Cobalt	EPA 6020B_(7/14)	4
Copper	EPA 6020B_(7/14)	4
Iron	EPA 6020B_(7/14)	4

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
Lead	EPA 6020B_(7/14)	4
Manganese	EPA 6020B_(7/14)	4
Molybdenum	EPA 6020B_(7/14)	4
Nickel	EPA 6020B_(7/14)	4
Selenium	EPA 6020B_(7/14)	4
Silver	EPA 6020B_(7/14)	4
Strontium	EPA 6020B_(7/14)	4
Thallium	EPA 6020B_(7/14)	4
Tin	EPA 6020B_(7/14)	
Vanadium	EPA 6020B_(7/14)	4
Zinc	EPA 6020B_(7/14)	4
Mercury, Liquid Waste	EPA 7470A_1_1994	2
Mercury, Solid Waste	EPA 7471B_(1/98)	4
Propylene glycol	EPA 8015C_(11/00)	
2,4'-DDD	EPA 8081B_(2/07)	4
2,4'-DDE	EPA 8081B_(2/07)	4
2,4'-DDT	EPA 8081B_(2/07)	4
4,4'-DDD	EPA 8081B_(2/07)	4
4,4'-DDE	EPA 8081B_(2/07)	4
4,4'-DDT	EPA 8081B_(2/07)	4
Alachlor	EPA 8081B_(2/07)	4
Aldrin	EPA 8081B_(2/07)	4
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081B_(2/07)	4
alpha-Chlordane	EPA 8081B_(2/07)	4
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081B_(2/07)	4
Chlordane (tech.)	EPA 8081B_(2/07)	4
delta-BHC	EPA 8081B_(2/07)	4
Dieldrin	EPA 8081B_(2/07)	4
Endosulfan I	EPA 8081B_(2/07)	4
Endosulfan II	EPA 8081B_(2/07)	4
Endosulfan sulfate	EPA 8081B_(2/07)	4
Endrin	EPA 8081B_(2/07)	4
Endrin aldehyde	EPA 8081B_(2/07)	4
Endrin ketone	EPA 8081B_(2/07)	4
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081B_(2/07)	4
gamma-Chlordane	EPA 8081B_(2/07)	4
Heptachlor	EPA 8081B_(2/07)	4

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
Heptachlor epoxide	EPA 8081B_(2/07)	4
Hexachlorobenzene	EPA 8081B_(2/07)	
Isodrin	EPA 8081B_(2/07)	4
Methoxychlor	EPA 8081B_(2/07)	4
Mirex	EPA 8081B_(2/07)	4
Permethrin (total)	EPA 8081B_(2/07)	
Toxaphene (Chlorinated camphene)	EPA 8081B_(2/07)	4
trans-Nonachlor	EPA 8081B_(2/07)	4
2,2', 3,3', 4,4', 5-Heptachlorobiphenyl	EPA 8082A_(2/07)	4
2,2', 3,3',4,4'-Hexachlorobiphenyl	EPA 8082A_(2/07)	4
2,2', 3,4', 5,5', 6-Heptachlorobiphenyl	EPA 8082A_(2/07)	4
2,2', 3,4,4', 5'-Hexachlorobiphenyl	EPA 8082A_(2/07)	4
2,2', 3,5'-Tetrachlorobiphenyl	EPA 8082A_(2/07)	4
2,2', 4,5,5'-Pentachlorobiphenyl	EPA 8082A_(2/07)	4
2,2', 5-Trichlorobiphenyl	EPA 8082A_(2/07)	4
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)	EPA 8082A_(2/07)	4
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)	EPA 8082A_(2/07)	4
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ-180)	EPA 8082A_(2/07)	4
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ-153)	EPA 8082A_(2/07)	4
2,2',5,5'-Tetrachlorobiphenyl (BZ-52)	EPA 8082A_(2/07)	4
2,3', 4,4'-Tetrachlorobiphenyl	EPA 8082A_(2/07)	4
2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)	EPA 8082A_(2/07)	4
2,4,4'-Trichlorobiphenyl (BZ-28)	EPA 8082A_(2/07)	4
2,4'-Dichlorobiphenyl (BZ-8)	EPA 8082A_(2/07)	4
2-Chlorobiphenyl (BZ-1)	EPA 8082A_(2/07)	4
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)	EPA 8082A_(2/07)	4
3,3',4,4'-Tetrachlorobiphenyl (BZ-77)	EPA 8082A_(2/07)	4
Aroclor-1016 (PCB-1016)	EPA 8082A_(2/07)	4
Aroclor-1221 (PCB-1221)	EPA 8082A_(2/07)	4
Aroclor-1232 (PCB-1232)	EPA 8082A_(2/07)	4
Aroclor-1242 (PCB-1242)	EPA 8082A_(2/07)	4
Aroclor-1242 (PCB-1242)	EPA 8082A_(2/07)	4
Aroclor-1248 (PCB-1248)	EPA 8082A_(2/07)	4
Aroclor-1254 (PCB-1254)	EPA 8082A_(2/07)	4
Aroclor-1260 (PCB-1260)	EPA 8082A_(2/07)	4
Aroclor-1262 (PCB-1262)	EPA 8082A_(2/07)	4
Aroclor-1268 (PCB-1268)	EPA 8082A_(2/07)	4

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
2,4,5-T	EPA 8151A_(1/98)	4
2,4-D	EPA 8151A_(1/98)	4
2,4-DB	EPA 8151A_(1/98)	4
Dalapon	EPA 8151A_(1/98)	4
Dicamba	EPA 8151A_(1/98)	4
Dichloroprop (Dichlorprop)	EPA 8151A_(1/98)	4
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151A_(1/98)	4
MCPA	EPA 8151A_(1/98)	4
MCPP	EPA 8151A_(1/98)	4
Pentachlorophenol	EPA 8151A_(1/98)	
Silvex (2,4,5-TP)	EPA 8151A_(1/98)	4
Organo-tins	Krone 1988	
Diesel range organics (DRO)	WDOE NWTPH-Dx_(1997)	4
Gasoline range organics (GRO)	WDOE NWTPH-Gx_(1997)	4
1H,1H,2H,2H,-Perfluorodecanesulfonic acid (8:2 FTS)	ALS LCP-PFC Rev. 11.0	4
1H,1H,2H,2H,-Perfluorooctansulfonic acid (6:2 FTS)	ALS LCP-PFC Rev. 11.0	4
N-Ethylperfluorooctane sulfonamide (EtFOSA)	ALS LCP-PFC Rev. 11.0	4
N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA)	ALS LCP-PFC Rev. 11.0	4
N-Ethylperfluorooctanesulfonamidoethanol (EtFOSE)	ALS LCP-PFC Rev. 11.0	4
N-Methylperfluorooctane sulfonamide (MeFOSA)	ALS LCP-PFC Rev. 11.0	4
N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA)	ALS LCP-PFC Rev. 11.0	4
N-Methylperfluorooctanesulfonamido ethanol (MeFOSE)	ALS LCP-PFC Rev. 11.0	4
Perfluorobutane sulfonic acid (PFBS)	ALS LCP-PFC Rev. 11.0	4
Perfluorodecane sulfonic acid (PFDS)	ALS LCP-PFC Rev. 11.0	4
Perfluorodecanoic acid (PFDA)	ALS LCP-PFC Rev. 11.0	4
Perfluorododecanoic acid (PFDoA)	ALS LCP-PFC Rev. 11.0	4
Perfluoroheptane sulfonic acid (PFHpS)	ALS LCP-PFC Rev. 11.0	4
Perfluoroheptanoic acid (PFHpA)	ALS LCP-PFC Rev. 11.0	4
Perfluorohexane sulfonic acid (PFHxS)	ALS LCP-PFC Rev. 11.0	4
Perfluorohexanoic acid (PFHxA)	ALS LCP-PFC Rev. 11.0	4
Perfluorononanoic acid (PFNA)	ALS LCP-PFC Rev. 11.0	4
Perfluorooctane sulfonamide (PFOSA)	ALS LCP-PFC Rev. 11.0	4
Perfluorooctane sulfonic acid (PFOS)	ALS LCP-PFC Rev. 11.0	4
Perfluorooctanoic acid (PFOA)	ALS LCP-PFC Rev. 11.0	4
Perfluoropentanoic acid (PFPeA)	ALS LCP-PFC Rev. 11.0	4
Perfluorotetradecanoic acid (PFTeDA)	ALS LCP-PFC Rev. 11.0	4
Perfluorotridecanoic acid (PFTrDA)	ALS LCP-PFC Rev. 11.0	4

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
Perfluoroundecanoic acid (PFUnA)	ALS LCP-PFC Rev. 11.0	4
1,1,1,2-Tetrachloroethane	EPA 8260D_4_(6/18)	6,12
1,1,1-Trichloro-2,2,2-trifluoroethane	EPA 8260D_4_(6/18)	6,12
1,1,1-Trichloroethane	EPA 8260D_4_(6/18)	6,12
1,1,2,2-Tetrachloroethane	EPA 8260D_4_(6/18)	6,12
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA 8260D_4_(6/18)	6,12
1,1,2-Trichloroethane	EPA 8260D_4_(6/18)	6,12
1,1-Dichloroethane	EPA 8260D_4_(6/18)	6,12
1,1-Dichloroethylene	EPA 8260D_4_(6/18)	6,12
1,1-Dichloropropene	EPA 8260D_4_(6/18)	6,12
1,2,3-Trichlorobenzene	EPA 8260D_4_(6/18)	6,12
1,2,3-Trichloropropane	EPA 8260D_4_(6/18)	6,12
1,2,4-Trichlorobenzene	EPA 8260D_4_(6/18)	6,12
1,2,4-Trimethylbenzene	EPA 8260D_4_(6/18)	6,12
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260D_4_(6/18)	6,12
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260D_4_(6/18)	6,12
1,2-Dichlorobenzene	EPA 8260D_4_(6/18)	6,12
1,2-Dichloroethane (Ethylene dichloride)	EPA 8260D_4_(6/18)	6,12
1,2-Dichloropropane	EPA 8260D_4_(6/18)	6,12
1,3,5-Trimethylbenzene	EPA 8260D_4_(6/18)	6,12
1,3-Dichlorobenzene	EPA 8260D_4_(6/18)	6,12
1,3-Dichloropropane	EPA 8260D_4_(6/18)	6,12
1,4-Dichlorobenzene	EPA 8260D_4_(6/18)	6,12
1,4-Dioxane (1,4- Diethyleneoxide)	EPA 8260D_4_(6/18)	6,12
1-Chlorohexane	EPA 8260D_4_(6/18)	6,12
2,2-Dichloropropane	EPA 8260D_4_(6/18)	6,12
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260D_4_(6/18)	6,12
2-Chloroethyl vinyl ether	EPA 8260D_4_(6/18)	6,12
2-Chlorotoluene	EPA 8260D_4_(6/18)	6,12
2-Hexanone	EPA 8260D_4_(6/18)	6,12
2-Nitropropane	EPA 8260D_4_(6/18)	6,12
4-Bromofluorobenzene	EPA 8260D_4_(6/18)	6,12
4-Chlorotoluene	EPA 8260D_4_(6/18)	6,12
4-Isopropyltoluene (p-Cymene)	EPA 8260D_4_(6/18)	6,12
4-Methyl-2-pentanone (MIBK)	EPA 8260D_4_(6/18)	6,12
Acetone	EPA 8260D_4_(6/18)	6,12
Acetonitrile	EPA 8260D_4_(6/18)	6,12

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
Acrolein (Propenal)	EPA 8260D_4_(6/18)	6,12
Acrylonitrile	EPA 8260D_4_(6/18)	6,12
Allyl chloride (3-Chloropropene)	EPA 8260D_4_(6/18)	6,12
Benzene	EPA 8260D_4_(6/18)	6,12
Bromobenzene	EPA 8260D_4_(6/18)	6,12
Bromochloromethane	EPA 8260D_4_(6/18)	6,12
Bromodichloromethane	EPA 8260D_4_(6/18)	6,12
Bromoform	EPA 8260D_4_(6/18)	6,12
Carbon disulfide	EPA 8260D_4_(6/18)	6,12
Carbon tetrachloride	EPA 8260D_4_(6/18)	6,12
Chlorobenzene	EPA 8260D_4_(6/18)	6,12
Chlorodibromomethane	EPA 8260D_4_(6/18)	6,12
Chloroethane (Ethyl chloride)	EPA 8260D_4_(6/18)	6,12
Chloroform	EPA 8260D_4_(6/18)	6,12
Chloroprene (2-Chloro-1,3-butadiene)	EPA 8260D_4_(6/18)	6,12
cis & trans-1,2-Dichloroethene	EPA 8260D_4_(6/18)	6,12
cis-1,2-Dichloroethylene	EPA 8260D_4_(6/18)	6,12
cis-1,3-Dichloropropene	EPA 8260D_4_(6/18)	6,12
cis-1,4-Dichloro-2-butene	EPA 8260D_4_(6/18)	6,12
Dibromochloropropane	EPA 8260D_4_(6/18)	6,12
Dibromomethane	EPA 8260D_4_(6/18)	6,12
Dichlorodifluoromethane (Freon-12)	EPA 8260D_4_(6/18)	6,12
Diethyl ether	EPA 8260D_4_(6/18)	6,12
Ethyl acetate	EPA 8260D_4_(6/18)	6,12
Ethyl methacrylate	EPA 8260D_4_(6/18)	6,12
Ethyl tert-Butyl alcohol	EPA 8260D_4_(6/18)	6,12
Ethylbenzene	EPA 8260D_4_(6/18)	6,12
Hexachlorobutadiene	EPA 8260D_4_(6/18)	6,12
Iodomethane (Methyl iodide)	EPA 8260D_4_(6/18)	6,12
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260D_4_(6/18)	6,12
Isopropylbenzene	EPA 8260D_4_(6/18)	6,12
m+p-xylene	EPA 8260D_4_(6/18)	6,12
Methacrylonitrile	EPA 8260D_4_(6/18)	6,12
Methyl bromide (Bromomethane)	EPA 8260D_4_(6/18)	6,12
Methyl chloride (Chloromethane)	EPA 8260D_4_(6/18)	6,12
Methyl tert-butyl ether (MTBE)	EPA 8260D_4_(6/18)	6,12
Methylene chloride (Dichloromethane)	EPA 8260D_4_(6/18)	6,12

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
Naphthalene	EPA 8260D_4_(6/18)	6,12
n-Butylbenzene	EPA 8260D_4_(6/18)	6,12
n-Propylbenzene	EPA 8260D_4_(6/18)	6,12
o-Xylene	EPA 8260D_4_(6/18)	6,12
sec-Butylbenzene	EPA 8260D_4_(6/18)	6,12
Styrene	EPA 8260D_4_(6/18)	6,12
tert-amylmethylether (TAME)	EPA 8260D_4_(6/18)	6,12
tert-Butyl alcohol	EPA 8260D_4_(6/18)	6,12
tert-Butylbenzene	EPA 8260D_4_(6/18)	6,12
Tetrachloroethylene (Perchloroethylene)	EPA 8260D_4_(6/18)	6,12
Toluene	EPA 8260D_4_(6/18)	6,12
trans-1,2-Dichloroethylene	EPA 8260D_4_(6/18)	6,12
trans-1,3-Dichloropropylene	EPA 8260D_4_(6/18)	6,12
trans-1,4-Dichloro-2-butene	EPA 8260D_4_(6/18)	6,12
Trichloroethene (Trichloroethylene)	EPA 8260D_4_(6/18)	6,12
Trichlorofluoromethane (Freon 11)	EPA 8260D_4_(6/18)	6,12
Vinyl acetate	EPA 8260D_4_(6/18)	6,12
Vinyl chloride	EPA 8260D_4_(6/18)	6,12
Xylene (total)	EPA 8260D_4_(6/18)	6,12
1,2,4,5-Tetrachlorobenzene	EPA 8270E_6_(6/18)	2,7,12
1,2,4-Trichlorobenzene	EPA 8270E_6_(6/18)	7,12
1,2-Dichlorobenzene	EPA 8270E_6_(6/18)	7,12
1,2-Diphenylhydrazine	EPA 8270E_6_(6/18)	7,12
1,3-Dichlorobenzene	EPA 8270E_6_(6/18)	7,12
1,4-Dichlorobenzene	EPA 8270E_6_(6/18)	7,12
2,3,4,6-Tetrachlorophenol	EPA 8270E_6_(6/18)	7,12
2,4,5-Trichlorophenol	EPA 8270E_6_(6/18)	7,12
2,4,6-Trichlorophenol	EPA 8270E_6_(6/18)	7,12
2,4-Dichlorophenol	EPA 8270E_6_(6/18)	7,12
2,4-Dimethylphenol	EPA 8270E_6_(6/18)	7,12
2,4-Dinitrophenol	EPA 8270E_6_(6/18)	7,12
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270E_6_(6/18)	7,12
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270E_6_(6/18)	7,12
2-Chloronaphthalene	EPA 8270E_6_(6/18)	7,12
2-Chlorophenol	EPA 8270E_6_(6/18)	7,12
2-Methylnaphthalene	EPA 8270E_6_(6/18)	7,12
2-Methylphenol (o-Cresol)	EPA 8270E_6_(6/18)	7,12

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
2-Nitroaniline	EPA 8270E_6_(6/18)	7,12
2-Nitrophenol	EPA 8270E_6_(6/18)	7,12
3,3'-Dichlorobenzidine	EPA 8270E_6_(6/18)	7,12
3-Nitroaniline	EPA 8270E_6_(6/18)	7,12
4,6-Dinitro-2-methylphenol	EPA 8270E_6_(6/18)	7,12
4-Bromophenyl phenyl ether (BDE-3)	EPA 8270E_6_(6/18)	7,12
4-Chloro-3-methylphenol	EPA 8270E_6_(6/18)	7,12
4-Chloroaniline	EPA 8270E_6_(6/18)	7,12
4-Chlorophenyl phenylether	EPA 8270E_6_(6/18)	7,12
4-Nitroaniline	EPA 8270E_6_(6/18)	7,12
4-Nitrophenol	EPA 8270E_6_(6/18)	7,12
Acenaphthene	EPA 8270E_6_(6/18)	7,12
Acenaphthylene	EPA 8270E_6_(6/18)	7,12
Acetophenone	EPA 8270E_6_(6/18)	7,12
Aniline	EPA 8270E_6_(6/18)	7,12
Anthracene	EPA 8270E_6_(6/18)	7,12
Atrazine	EPA 8270E_6_(6/18)	7,12
Benzidine	EPA 8270E_6_(6/18)	7,12
Benzo(a)anthracene	EPA 8270E_6_(6/18)	7,12
Benzo(a)pyrene	EPA 8270E_6_(6/18)	7,12
Benzo(g,h,i)perylene	EPA 8270E_6_(6/18)	7,12
Benzo(k)fluoranthene	EPA 8270E_6_(6/18)	7,12
Benzo[b]fluoranthene	EPA 8270E_6_(6/18)	7,12
Benzoic acid	EPA 8270E_6_(6/18)	7,12
Benzyl alcohol	EPA 8270E_6_(6/18)	7,12
Biphenyl	EPA 8270E_6_(6/18)	7,12
bis(2-Chloroethoxy)methane	EPA 8270E_6_(6/18)	7,12
bis(2-Chloroethyl) ether	EPA 8270E_6_(6/18)	7,12
bis(2-Chloroisopropyl) ether	EPA 8270E_6_(6/18)	7,12
Butyl benzyl phthalate	EPA 8270E_6_(6/18)	7,12
Carbazole	EPA 8270E_6_(6/18)	7,12
Chrysene	EPA 8270E_6_(6/18)	7,12
Di(2-ethylhexyl)phthalate	EPA 8270E_6_(6/18)	7,12
Dibenzofuran	EPA 8270E_6_(6/18)	7,12
Diethyl phthalate	EPA 8270E_6_(6/18)	7,12
Dimethyl phthalate	EPA 8270E_6_(6/18)	7,12
Di-n-butyl phthalate	EPA 8270E_6_(6/18)	7,12

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
Di-n-octyl phthalate	EPA 8270E_6_(6/18)	7,12
Fluoranthene	EPA 8270E_6_(6/18)	7,12
Fluorene	EPA 8270E_6_(6/18)	7,12
Hexachlorobutadiene	EPA 8270E_6_(6/18)	7,12
Hexachlorocyclopentadiene	EPA 8270E_6_(6/18)	7,12
Hexachloroethane	EPA 8270E_6_(6/18)	7,12
Indeno(1,2,3-cd) pyrene	EPA 8270E_6_(6/18)	7,12
Isophorone	EPA 8270E_6_(6/18)	7,12
m+p Cresol	EPA 8270E_6_(6/18)	7,12
Naphthalene	EPA 8270E_6_(6/18)	7,12
Nitrobenzene	EPA 8270E_6_(6/18)	7,12
N-Nitrosodimethylamine	EPA 8270E_6_(6/18)	7,12
N-Nitroso-di-n-propylamine	EPA 8270E_6_(6/18)	7,12
N-Nitrosodiphenylamine	EPA 8270E_6_(6/18)	7,12
Pentachlorophenol	EPA 8270E_6_(6/18)	7,12
Phenanthrene	EPA 8270E_6_(6/18)	7,12
Phenol	EPA 8270E_6_(6/18)	7,12
Pyrene	EPA 8270E_6_(6/18)	7,12
Pyridine	EPA 8270E_6_(6/18)	7,12
1,4-Dioxane (1,4- Diethyleneoxide)	EPA 8270E_6_(6/18) SIM	13
1-Methylnaphthalene	EPA 8270E_6_(6/18) SIM	13
2-Methylnaphthalene	EPA 8270E_6_(6/18) SIM	13
Acenaphthene	EPA 8270E_6_(6/18) SIM	13
Acenaphthylene	EPA 8270E_6_(6/18) SIM	13
Anthracene	EPA 8270E_6_(6/18) SIM	13
Benzo(a)anthracene	EPA 8270E_6_(6/18) SIM	13
Benzo(a)pyrene	EPA 8270E_6_(6/18) SIM	13
Benzo(g,h,i)perylene	EPA 8270E_6_(6/18) SIM	13
Benzo(k)fluoranthene	EPA 8270E_6_(6/18) SIM	13
Benzo[b]fluoranthene	EPA 8270E_6_(6/18) SIM	13
Chrysene	EPA 8270E_6_(6/18) SIM	13
Dibenz(a,h) anthracene	EPA 8270E_6_(6/18) SIM	13
Dibenzofuran	EPA 8270E_6_(6/18) SIM	13
Fluoranthene	EPA 8270E_6_(6/18) SIM	13
Fluorene	EPA 8270E_6_(6/18) SIM	13
Indeno(1,2,3-cd) pyrene	EPA 8270E_6_(6/18) SIM	13
Naphthalene	EPA 8270E_6_(6/18) SIM	13

Matrix/Analyte	Method	Notes
Solid and Chemical Materials		
Pentachlorophenol	EPA 8270E_6_(6/18) SIM	13
Phenanthrene	EPA 8270E_6_(6/18) SIM	13
Pyrene	EPA 8270E_6_(6/18) SIM	13
Carbaryl (Sevin)	EPA 8321B_2_(2/07)	4
Fecal coliform-count	SM 9221 B+E1+C (LTB/BGB/EC-MPN)	2
Total coliforms-count	SM 9222 B (mEndo)	2
Fecal coliform-count	SM 9222 D (mFC)-06	2
Particle Size Distribution	ASTM D422	4,15
Ignitability	EPA 1020A_1_1992	4
Particle Size Distribution	PSEP 1986 Wet Sieve	

Accredited Parameter Note Detail

(1) Accreditation does not apply to compliance testing due to the sample holding time requirement for pH. (2) Accrediltation is limited to liquid matrix only. (3) Interim accreditation pending the successful completion of an on-site audit to verify method capabilities (WAC 173-50-100). (4) Accreditation based in part on recognition of Oregon NELAP accreditation. (5) Provisional accreditation pending submittal of acceptable Proficiency Testing (PT) results (WAC 173-50-110). (6) Provisional accreditation pending laboratory update from EPA Method 8260C to the new method EPA 8260 and pending acceptable audit corrective actions. (7) Provisional accreditation pending laboratory update from EPA Method 8270D to the new method EPA 8270E and pending acceptable audit corrrective actions. (8) Provisional accreditation pending laboratory update from EPA Method 608 to the new method EPA 608.3 and pending acceptable audit corrective actions. (9) Provisional accreditation pending laboratory update from EPA Method 624 to the new method EPA 624.1 and pending aceptable audit corrective actions. (10) Provisional accreditation pending laboratory update from EPA Method 625 to the new method EPA 625.1 and pending acceptable audit corrective actions. (11) Provisional accreditation pending completion of audit corrective actions. (12) Accreditation based in part on recognition of DoD-ELAP accreditation.(13) Provisional accreditation pending laboratory update to the new method EPA 8270E SIM and pending acceptable audit corrrective actions.(14) Screening (15) Provisional accreditation pending submittal of acceptable corrective action report since a PT is no longer available for this method.

Rebecca Wood

Authentication Signature

Rebecca Wood, Lab Accreditation Unit Supervisor

07/20/2022

Date

Nitrogen, Total and Soluble Kjeldahl

DOCUMENT ID: GEN-TKN, REV 17.0

Prepared By: Inorganics Manager, Rachel Moore
Signature on File

Approved By: Quality Assurance Manager, Kurt Clarkson
Signature on File

Approved By: Laboratory Director, Todd Poyfair
Signature on File

1) Scope & Applicability

1.1 This procedure is used to determine Total Kjeldahl Nitrogen (TKN) in drinking water, wastewater, and surface water using methodology described in ASTM Methods ASTM D3590-17A and D1426-15B and EPA 351.4 (other equivalent versions of the same methods may be used for specified reporting purposes). The procedure is also applicable to domestic and industrial waste matrices.

1.2 The procedure also describes options for determination of Soluble Kjeldahl Nitrogen (SKN).

1.3 With modifications described in this SOP, soils and certain solids can be analyzed.

1.4 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DOD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management,* may supersede the requirements defined in this SOP.

2) Summary of Procedure

2.1 Organic and ammonia nitrogen are determined by converting organic nitrogen to ammonia using digestion with hot concentrated sulfuric acid, catalyzed by copper sulfate. Ammonia is then measured using ion-specific electrode. It is recognized that background subtraction is commonly used for TKN analyses however; background subtraction is not included in ASTM D3590-17A. If the ASTM D1426-15B ISE option is used, background subtraction is not performed.

2.2 The Method Reporting Limit (MRL) for water is 0.2mg/L and 40mg/kg for soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL). The Method Detection Limit (MDL) in water has been determined at 0.08mg/L. The soil MDL has been determined at 8mg/kg.

3) Definitions

3.1 For laboratory definitions applicable to most analyses, refer to the SOP for *Sample Batches*.

4) Responsibilities

4.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

4.2 It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the ALS-Kelso *SOP* for *Employee Training and Orientation* (ADM-TRAIN), is also the responsibility of the department supervisor/manager.

5) Interferences

5.1 Interferences from metals are minimized with the addition of NaI.

5.2 High nitrate concentrations (>10 times TKN level) result in low TKN values. This can be prevented by the use of the anion exchange resin (chloride form) to remove the nitrate prior to TKN analysis.

6) Safety

6.1 All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personal protective equipment, such as, safety glasses, lab coat and the correct gloves.

6.2 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDSs prior to beginning this method.

6.3 Sodium Hydroxide (NaOH) is a strong caustic and a severe health and contact hazard. Use nitrile or latex gloves while handling pellets or preparing solutions.

6.4 This procedure involves the use of concentrated sulfuric acid. Care must be taken to prevent direct contact with concentrated sulfuric acid.

6.5 During digestion, SO_3 fumes are produced. Ensure that the apparatus is set up properly.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Sample bottles should be plastic and must be thoroughly cleaned and rinsed prior to use. Soil samples may be collected in glass jars.

7.2 A minimum of 500mL of sample should be collected, preferably 1L. For soil samples, a minimum of 10g should be collected. Collecting 8 oz. jars of soil improves subsampling homogeneity.

7.3 Water samples are to be preserved with H_2SO_4 to a pH <2 at time of collection.

7.4 Samples must be stored refrigerated at ≤6°C and analyzed within 28 days from date of sample collection.

8) Standards, Reagents, and Consumable Materials

8.1 Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking (ADM-RLT)* for the complete procedure and documentation requirements.

8.2 All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I or ASTM Type II reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

8.3 Reagents and Standards must comply with the traceability, labeling and documentation practices specified in the SOPs: *Making Entries onto Analytical Records* and *Reagent and Standards Login and Tracking* (ADM-RLT).

8.4 10N NaOH: Dilute 400g Sodium Hydroxide with water to one liter in a volumetric flask. This reagent is good for 6 months.

8.5 Copper sulfate digestion solution: Dissolve 3.65g $CuSO_4$ in 300mL of DI water and 67mL conc. H_2SO_4. Dissolve 67g K_2SO_4 in same solution and bring to 500mL with DI water in a volumetric flask. Prepare daily.

8.6 Ammonia stock standard 10,000mg/L: Dissolved 38.1900g Ammonium Chloride (NH4Cl) with DI water to one Liter in a volumetric flask. (1.0mL = 10mg Ammonia nitrogen) This standard is good for 6 months.

8.7 1000mg/L Ammonia working standard solution: Dilute 100mL of stock solution with water to 1L in a volumetric flask. This standard is good for 6 months.

8.8 100mg/L Ammonia working standard solution: Dilute 10mL of stock solution with water to one Liter in a volumetric flask. This standard is good for 6 months.

8.9 10mg/L Ammonia working standard solution: Dilute one 1mL of stock solution with water to one Liter in a volumetric flask. This standard is good for 6 months.

8.10 1.0mg/L Ammonia working standard solution: Dilute one 0.1mL of stock solution with water to one Liter in a volumetric flask. This standard is good for 6 months.

8.11 1000mg/L Ammonia Nitrogen spiking solution: Dissolve 5.3391g Glycine with DI water to one Liter in a volumetric flask. This standard is good for 6 months.

8.12 NaOH/Sodium Thiosulfate Solution - Dissolve 500g NaOH in 800mL reagent water. Add 25g of Sodium Thiosulfate (NaS2O3 ‘ 5H20). Mix and bring to 1L with reagent water.

9) Apparatus and Equipment

9.1 Digestion apparatus: A Kjeldahl block digestion apparatus with 50mL digestion tubes and water

9.2 Orion Star A214

9.3 Orion Ammonia Electrode

9.4 150mL Beakers, Volumetric Flasks

9.5 Stir Plate and stir bars

10) Preventative Maintenance

10.1 All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described herein. The entry in the log must include: date of event, the initials of who performed the work, corrective action and a reference to analytical control.

10.2 Electrode preparation

10.2.1 Unscrew top and remove the glass electrode inner body from the electrode outer body. Set cap and inner body aside.

10.2.2 Remove bottom cap from electrode outer body.

10.2.3 Using tweezers, carefully grasp a white membrane from between paper separators.

10.2.4 Place membrane against threads of electrode outer body. Membrane should have no wrinkles.

10.2.5 Place the loose membrane cap over the ends and screw on until finger tight.

10.2.6 Squeeze internal filling solution at optimum results into the outer electrode body up to line.

10.2.7 Place inner body into outer body containing internal filling solution and screw on upper cap.

10.2.8 Shake the completely assembled probe to remove bubbles. For best results, soak probe overnight in filling solution.

10.2.9 Membrane failure may be apparent on visual inspection as dark spots or discoloration of the membrane. Change membrane weekly, even if spots do not appear.

10.2.10 Store the electrode in 1mg/L ammonia solution.

10.3 Wipe down the block digester after use to remove any residual acid.

11) Procedure

11.1 Digestion

11.1.1 For Soluble Kjeldahl Nitrogen (SKN) only, weigh out 5.0g of sample. Put into a 50mL centrifuge tube and bring to a 50mL volume with DI water. Shake the sample on the wrist action shaker for 5 minutes, followed by centrifuging for 5 minutes. Decant the supernatant and continue with the digestion procedure.

11.1.2 Place appropriate aliquot of sample (25mL liquid, 0.27 to 0.30g solid) in a 50mL Kjeldahl digestion tube. Add 10mL digestion solution to liquids and 20mL to solids. Add spiking solution to the designated QC samples (spike waters and solids with 0.5mL and 1mL respectively). Add approximately 5 boiling chips and 5 drops of antifoam to each tube and place on block.

11.1.3 The HotBlock® TKN unit is pre-set to 160˚C. This is approximately the temperature most frequently used in the initial digestion phase of TKN analysis.

NOTE: The set point of the block will not be the same as the temperature of the liquids being digested, as this will vary according to outside factors.

11.1.4 Simple Manual Controller:

- Turn on the controller. It will preheat to 160˚C.
- When the temperature stabilizes at 160˚C, press the green button.
- The display will read “nonE”. Use the up arrow to show “ProF”.
- Press the green button twice. The ramp indicator light will show that a ramp/soak profile is active. This will follow the temperature and time profile.

11.1.5 Take note of start time. After 1 hour, cap all samples. At this time, the block will ramp up to 380˚C over the next hour and a half, for a total of 2.5 hour digestion time. When samples have digested for 2.5 hours, put samples on a rack, remove caps, and let cool for 3-4 minutes. Add about 30mL DI water to tubes and transfer to 50mL centrifuge tubes. Bring volume up to 45mL.

11.1.6 When seawater samples are to be digested, use a 10mL aliquot of sample instead of the 25mL normally used for fresh, non-saline water samples.

11.1.7 Place the 10mL seawater aliquot in a 50mL Kjeldahl digestion tube; add 20mL of digestion solution.

11.2 Distillation

11.2.1 Distillation of sample digest may be necessary in order to comply with NPDES (40 CFR) wastewater regulatory requirements. However, digest distillation is not absolutely necessary for ammonia quantification by ion selective electrode. Analyst should consult with their supervisor prior to sample analysis to determine appropriate analytical procedure.

11.2.2 Distillation Procedure

11.2.2.1 Adjust the pH of a 45mL sample aliquot to pH 9.5. Add 6N NaOH/Sodium Thiosulfate solution to the sample to adjust the sample pH to 9.5 (about 5mL). Mix sample. Check the sample pH with a narrow range pH paper.

11.2.2.2 Place the pH adjusted sample in a boiling tube. Add 2-3 micro-porous boiling stones and 0.5 milliners antifoam solution. Assemble the distillation apparatus in the heater block. Lubricate ground glass joints of distillation apparatus with silicone grease (Lubriseal) to ensure that the glass doesn't fuse together.

11.2.2.3 Place 2.5mL of receiving solution (0.04N H2SO4 solution) in a 50mL centrifuge tube. Place the centrifuge tube in the base slot of the heating block. Select the receiver solution according to analysis method to be used on distillate.

11.2.2.4 Place the condenser stem in the centrifuge tube, slide cylinder and stem under condenser, and connect stem to condenser with WestClip. When assembled, the condenser tip of the distillation apparatus will be submerged in the receiving solution contained in the centrifuge tube.

11.2.2.5 Heat the distillation apparatus - Set heater block temperature controls to the following settings:

RATE1: 15 ˚C/min	RATE2: 0
TEMP1: 210 ˚C	TEMP2: 210˚C
TIME1: 2.0 Hour (max)	TIME2: 0

Timing Configuration: CONF1

11.2.2.6 Collect a minimum of 40mL of distillate.

11.2.2.7 Discontinue heating. Remove the stopper from the top of the "T"-joint. The stopper removal is needed to prevent the collected distillate from being sucked back into the apparatus during cooling.

11.2.2.8 Bring distillate to 50mL final volume with reagent water.

11.3 The digestate is analyzed for Ammonia using an approved method. Routinely, this is Ion Selective Electrode (ISE) using ASTM D1426-15B or Standard Methods 4500-NH3 E. Refer to SOP GEN-4500NH3-E for the Standard Methods procedure. Alternatively, Ammonia may be determined by flow injection analysis methods EPA 350.1 and SM 4500 4500-NH3 G (ALS SOP GEN-350.1). ASTM D1426-15B is the default procedure and is provided below.

11.4 Ion Selective Electrode Analysis - Thermo Scientific Orion Star A214.

11.4.1 Initial Instrument Set Up – Refer to Orion Star A214 reference manual.

11.4.1.1 G:\QA Controlled Documents\Lab SOPs\GEN\ISE MANUAL.pdf.

11.4.2 Standardization - Refer to Chapter 5 of the ISE Manual.

11.4.2.1 Prepare calibration curves with three standard solutions bracketing the expected concentrations of the samples. The meter is standardized based on the mV readings. Using the following functions, the meter records the mV response for the individual standards. **A multi-point linear calibration model is not used.**

11.4.2.2 Prepare 1mg/L, 10mg/L, and 100mg/L standard solutions from 10,000mg/L NH4Cl stock solution.

11.4.2.3 Standardize - Refer to Chapter 5 of ISE Manual:

G:\QA Controlled Documents\Lab SOPs\GEN\ISE MANUAL.pdf

11.4.2.4 The slope must be between -54.0 to -60.0mV.

11.4.2.5 The slope will vary depending on temperature (usually -57 to -59mV).

11.4.3 Initial Calibration Verification Analysis

11.4.3.1 Put 50mL of 12ppm NH3 standard into a conductivity jar. Insert ammonia electrode into the solution. Add 1mL 10N NaOH. When stable, meter will say ready. Record result on benchsheet.

11.4.4 Initial Calibration Blank Analysis

11.4.4.1 Put 50mL of deionized water into a conductivity jar. Insert ammonia electrode into the solution. Add 1mL 10N NaOH. When stable, meter will say ready. Record result on benchsheet.

11.4.5 Continuing Calibration Verification Analysis

11.4.5.1 Put 50mL of 10ppm NH3 standard into a conductivity jar. Insert ammonia electrode into the solution. Add 1mL 10N NaOH. When stable, meter will say ready. Record result on benchsheet.

11.4.6 Method Blank Analysis

11.4.6.1 Put 50mL of digested MB and 1mL of 1N NaOH into a conductivity jar. Insert ammonia electrode into the solution. When stable, the meter will say ready. Record the results on the benchsheet.

11.4.7 LCS Analysis

11.4.7.1 Put 50mL of digested LCS and 1mL 1N NaOH into a conductivity jar. Insert ammonia electrode into the solution. When stable, the meter will say ready. Record the results on the benchsheet.

11.4.8 Sample/QC Analysis

11.4.8.1 Put 50mL of sample and 1mL 1N NaOH into a conductivity jar. Insert ammonia electrode into the solution. When stable, meter will say ready. Record the results on the benchsheet.

11.4.8.2 If the sample result falls below the low calibration standard, re-standardize the meter using solutions that bracket the expected result and re-analyze the sample. If the sample result exceeds the high calibration standard, dilute the sample and re-analyze.

12) QA/QC Requirements

12.1 Initial Precision and Recovery Validation

12.1.1 The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four analyte-free soil/sand samples are spiked with the LCS spike solution, then prepared and analyzed.

12.2 Method Detection Limits, LOD and LOQ:

12.2.1 Method detection limit (MDL), Limits of Detection (LOD) and Limits of Quantification (LOQ) must be determined before analysis of samples can begin. Refer to the SOP for *Performing and Documenting Method Detection Limit Studies and Estimation of Limits of Detection and Quantitation.*

12.3 The Method Reporting Limits (MRLs) used at ALS are the routinely reported lower limits of quantitation, which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which.

12.4 Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). For example projects managed under the DoD ELAP must follow requirements defined in the DoD *Quality Systems Manual for Environmental Laboratories.* General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management.* General QC Samples are:

12.4.1 Method Blank

12.4.1.1 A method blank is extracted and analyzed with every batch of 20 (or fewer) samples to monitor contamination during the analytical process. If the method blank shows any detections above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, and/or re-extraction and re-analysis. Specific project requirements and data quality objectives should be weighed against method blank results.

12.4.2 Lab Control Sample (LCS)

12.4.2.1 The LCS is composed of analyte-free solid matrix, which is spiked with the target analyte(s). The LCS is designed to monitor the performance of the procedure independent of matrix-related biases. The concentration of the spike in the LCS matrix should be at 5 to 10 times the MRL or at levels specified by a project specific quality analysis plan.

12.4.2.2 A lab control sample (LCS) must be prepared and analyzed with every batch of 20 (or fewer) samples. Calculate the LCS recovery as follows:

%R = X/TV x 100

Where: X = Concentration of the analyte recovered
TV = True value of amount spiked

12.4.2.3 The acceptance criteria are given in the ALS DQO Table. If the LCS fails acceptance criteria, corrective action must be taken. Corrective action includes recalculation, re-analysis, and/or re-extraction and re-analysis.

12.4.3 Matrix Spike

12.4.3.1 A matrix spike (MS) and duplicate matrix spike (DMS) must be prepared and analyzed with every batch of 20 (or fewer) samples. The MS/DMS is prepared by adding a known volume of the matrix spike solution to the sample and determining the spiked sample concentration. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where: X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

12.4.3.2 Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where R1= %recovery of the MS
R2= %recovery of the DMS

12.4.3.3 Following analysis of the MS, the percent recovery is calculated and compared to acceptance limits as referenced in the ALS Kelso DQO Table. If the recovery is within control limits, the results may be reported unqualified. If not, this indicates that the matrix potentially biases analyte recovery. Also verify that the spike level is at least five times the native concentration of the unspiked parent sample. If not, the results are reported with a qualifier that the background level is too high for accurate determination of recovery. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results, such as performing additional cleanups, dilution and re-analysis, or re-preparation and re-analysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

12.5 After the initial calibration of the instrument, the slope is determined and entered into the instrument. The slope will vary depending on the temperature (usually -57 to -59mV).

12.6 An ICV (Initial Calibration Verification) is analyzed at the start of each analytical batch. The acceptance criteria is ±10%. If outside this limit, the cause of the problem needs to be solved before proceeding.

12.7 An ICB (Initial Calibration Blank) is analyzed following the ICV. It must be less than 0.05mg/L.

12.8 A CCV (Continuing Calibration Verification) at 10mg/L is analyzed after the ICB and every ten readings and at the end of the run. It must be ±10%.

12.9 A CCB (Continuing Calibration Blank) is analyzed after each CCV. It must be less than 0.05mg/L.

13) Data Reduction and Reporting

13.1 Raw data and corrective actions must be recorded into the bench records by the analyst. All analysis descriptions (samples, blanks, CCVs, etc.), measurements, and readings are recorded on the TKN spreadsheet. The spreadsheet is used to document all analyses and to perform calculations leading for final sample results.

13.2 Use the spreadsheet described above to calculate sample concentrations. Calculate sample concentrations as follows:

Aqueous Samples:

$$Concentration\ (mg / L) = \frac{(Csol)\ (Vf)\ (D)}{(Vs)}$$

Where: Csol = Concentration in solution in mg/L
Vf = Final volume of digestate in L
D = Dilution factor
Vs = Volume of sample digested, liters

Non-aqueous Samples:

$$Concentration\ (mg / Kg) = \frac{(Csol)\ (Vf)\ (D)}{(W)}$$

Where: Csol = Concentration in solution in mg/L
Vf = Final volume of digestate in L
D = Dilution factor
W = Weight of sample digested in kg

13.3 Preliminary results are reviewed to determine if dilutions are required. Sample results to be reported are highlighted and reported directly from the spreadsheet. Concentration, dilution factor and sample identification number may be highlighted for reporting purposes. The benchsheet is signed and dated by the analyst.

13.4 It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified for samples (above). These results are then used to calculate QC determinations (recovery, RPD, etc.).

13.5 Reporting

13.5.1 Refer to the SOP for *Data Reporting and Report Generation* for reporting guidelines.

13.5.2 The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the ALS network directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS. Once the results are transferred, the report is reviewed.

13.5.3 Reports are generated in the ALS LIMS by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transferred to a file which Excel© uses to generate a report. The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.6 Data Review and Assessment

13.6.1 Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process (ADM-DREV)* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Manager to inclusion in the report narrative.

13.6.2 Bench sheets are completed and a batch lot number is assigned. The Manufacturer's lot numbers or ID's for the reagents are added to bench sheets.

13.7 Data Review and Assessment

13.7.1 Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP *Laboratory Data Review Process* (ADM-DREV) for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Manager for inclusion in the report narrative.

14) Contingencies for Handling Out-of-Control or Unacceptable Data

14.1 Refer to the SOP for *Nonconformance and Corrective Action Procedures* (ADM-NCAR) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2 Handling out-of-control or unacceptable data

14.2.1 On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, run logs, for example.

14.2.2 Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision.
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels.
- Sample holding time missed due to laboratory error or operations.
- Deviations from SOPs or project requirements.
- Laboratory analysis errors impacting sample or QC results.
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc.).
- Sample preservation or handling discrepancies due to laboratory or operations error.
- Customer inquiries concerning data quality or services (when applicable). NCAR not required for simple corrections with no impact to the client.
- Data errors reported to clients, non-conforming re-checks.
- Deficiencies found during internal or external audits.
- Login errors or shipping errors.
- IT issues if there is a significant impact to a client.
- Turnaround time complaints.

15) Method Performance

15.1 The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS aliquots are prepared and analyzed. The average percent recovery must meet the laboratory control sample acceptance limits.

15.2 The method detection limit (MDL) is established using the procedure described in the SOP *Performing and Documenting Method Detection Limit Studies and Estimation of Limits of Detection and Quantitation.* Method Reporting Limits are established for this method based on MDL studies.

16) Pollution Prevention and Waste Management

16.1 It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

16.2 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

16.3 This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS Kelso Chemical Hygiene Plan for details

16.4 This method uses a base. Waste base is hazardous to the sewer system and to the environment. All waste must be neutralized t prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS Kelso Chemical Hygiene Plan for details.

17) Training

17.1 Training outline

17.1.1 Review literature (see References section). Review the SOP. Also review safety procedures. Following these reviews, observe the procedure performed by an experienced analyst at least three times.

17.1.2 The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.3 Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to TNI's Initial Demonstration of Capability.

17.2 Training is documented following the *Employee Training and Orientation* (ADM-TRAIN).

17.2.1 When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18) Method Modifications

18.1 The reference method is modified to allow for the digestion of solid or semi-solids.

19) References

19.1 TNI Standard, Volume, 2009; 2016.

19.2 ISO/IEC 17025:2005, 2017.

19.3 DoD Quality Systems Manual for Environmental Laboratories, current version.

19.4 ASTM Methods D3590-17(A) and D1426-15B.

19.4.1 G:\QA\Methods\ASTM\D1426-2015.pdf.

19.5 EPA METHOD 351.4.

20) Changes since Last Revision

Revision Number	Effective Date	Document Editor	Description of Changes
16.0	5/6/2020	T. Caron	Reformatted SOP to current ALS branding. Minor typographical, grammatical, and formatting revisions to improve readability and consistency. Section 11: This section has been modified in its entirety to represent updated, cited methodologies.
17.0	4/6/2022	E. Davelaar	Updated SOP Signatories. Section 8.5: Added "digestion" between "cooper" and "sulfate".

			Removed section 8.12. Section 11.1: Updated in its entirety to reflect current practices. Section 11.2.2.1: Updated sample volume to 45mL and included approximate volume of NaOH/Sodium Thiosulfate to add. Section 11.2.2.2: Re-worded last sentence. Section 11.2.2.3: Updated concentration of H2SO4 to 0.04N. Removed sections 11.4.2.3, 11.4.2.5, 11.4.2.6, 11.4.2.7, and 11.4.2.8. New Sections: 11.4.2.4 and 11.4.2.5. Sections 11.4.6.1 and 11.4.7.1: Updated volume of MB and LCS to 50mL and volume of 1N NaOH to 1mL and added last sentence. Section 11.4.8.1: Updated volume of sample to 50mL and volume of 1N NaOH to 1mL.

21) Attachments, Tables, and Appendices

21.1 Table 1: Target Analytes – MRLs and MDLs.

21.2 Table 2: Summary of Corrective Actions.

TABLE 1
TARGET ANALYTES, MRLs, and MDLs

Analyte	Method Detection Limit		Method Reporting Limit	
	Water	Soil	Water	Soil
	ug/L	mg/kg	ug/L	mg/kg
Total Kjeldahl Nitrogen	0.08	8.0	0.2	40

TABLE 2

Summary of Corrective Actions

Method Reference	Control	Specification and Frequency	Acceptance Criteria	Corrective Action
ASTM 1426-15 EPA 351.4	ICV	After Standardization	± 10% (use statistical limit of 83-116% for Arizona samples)	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
ASTM 1426-15 EPA 351.4	ICB	Following ICV	<0.2mg/L	
ASTM 1426-15 EPA 351.4	CCV	After ICB, every 10 samples, and end of run	±10% Diff	Correct problem then repeat CCV or repeat ICAL
ASTM 1426-15 EPA 351.4	CCB	After each CCV	<0.2mg/L	Determine and correct source of contamination and reanalyze samples.
ASTM 1426-15 EPA 351.4	Method Blank	Include with each analysis batch (up to 20 samples)	<0.2mg/L	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20X amount in blank.
ASTM 1426-15 EPA 351.4	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	W: 72-129% S: 82-131%	If exceeds limits, re-extract and re-analyze
ASTM 1426-15 EPA 351.4	Matrix Spike	Include with each analysis batch (up to 20 samples)	±25%	Evaluate data to determine if the there is a matrix effect or analytical error
ASTM 1426-15 EPA 351.4	Sample Duplicates	Include with each analysis batch (up to 20 samples)	≤20% RPD	Re-homogenize and re-analyze if result is >5X the MRL

Ammonia by Flow Injection Analysis

DOCUMENT ID: GEN-350.1, REV 16.0

Prepared By: Inorganics Manager, Rachel Moore
Signature on File

Approved By: Quality Assurance Manager, Kurt Clarkson
Signature on File

Approved By: Laboratory Director, Todd Poyfair
Signature on File

1) Scope & Applicability

1.1 This procedure is used to determine the concentration of Ammonia in aqueous samples including drinking, ground, surface, and saline waters, and domestic and industrial wastes using EPA Method 350.1 and Standard Methods 4500 NH_3 G-2017. Ammonia in soil samples can be determined following extraction in a potassium chloride solution.

1.2 This method is applicable for the determination of ammonia concentrations in a range of 0.01mg/L to 5.0mg/L. The MRL for this test is 0.05mg/L and the MDL as been determined at 0.02mg/L. The MRL and MDL for low level ammonia analysis have been determined as 0.01mg/L and 0.003mg/L respectively.

1.3 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DOD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management,* may supersede the requirements defined in this SOP.

2) Summary of Procedure

2.1 For Regular level Ammonia analysis is performed using the Skalar Segmented Flow Analyzer. The Skalar's automated procedure for the determination of Ammonia / Total Nitrogen is based on the modified Berthelot reaction; ammonia buffered and dialyzed and is chlorinated to monochloramine which reacts with salicylate to 5-aminosalicylate. After oxidation and oxidative coupling a green colored complex is formed. The absorption of the formed complex is measured at 660nm.

2.2 Low level Ammonia analysis in water and the analysis of Ammonia in soil and sediments is performed using the Barn & Luebbe Segmented Flow Analyzer. Low level water are analyzed without distillation. Soil and sediment samples are extracted with 2M KCl using a wrist action shaker with extracts analyzed using the water procedure (based on the Plumb extraction procedure).

2.3 Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 660 nm, and is directly proportional to the original ammonia concentration in the sample.

2.4 Skalar Segmented Flow Analysis - The automated procedure for the determination of Ammonia / Total Nitrogen is based on the modified Berthelot reaction; ammonia buffered and dialyzed and is chlorinated to monochloramine which reacts with salicylate to 5-aminosalicylate. After oxidation and oxidative coupling a green colored complex is formed. The absorption of the formed complex is measured at 660nm.

3) Definitions

3.1 For laboratory definitions applicable to most analyses, refer to the SOP for *Sample Batches*.

4) Responsibilities

4.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

5) Interferences

5.1 Calcium and magnesium ions may precipitate if present in sufficient concentration. Tartrate or EDTA is added to the sample in-line in order to prevent this problem.

5.2 Color, turbidity and certain organic species may interfere. Turbidity is removed through manual filtration.

5.3 Over-acidic samples create a negative interference resulting in an inverted peak with samples that are at or near the method reporting limit.

5.4 Ensure that the pH of the dilution water and standard NH_3 solution approximates that of the sample.

5.5 Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out.

5.6 Residual chlorine must be removed by pre-treatment of the sample with sodium thiosulfate or other reagents before distillation.

6) Safety

6.1 All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personal protective equipment, such as, safety glasses, lab coat and the correct gloves.

6.2 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDSs prior to beginning this method.

6.3 Sodium Hydroxide (NaOH) is a strong caustic and a severe health and contact hazard. Use nitrile or latex gloves while handling pellets or preparing solutions.

6.4 Sulfuric acid (H_2SO_4) is a severe health, reactivity, and contact hazard. Always wear gloves and use caution when handling.

6.5 Phenol causes severe burns and is an extreme health hazard through skin absorption. Wear proper protective laboratory clothing including gloves and rinse any exposed skin IMMEDIATELY with copious amounts of water.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Water samples

7.1.1 Samples should be collected in plastic or glass bottles. Bottles must be

purchased as pre-cleaned containers or thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis.

7.1.2 Water samples are preserved immediately upon sampling or as soon as possible with H_2SO_4 to pH <2 and stored at ≤6°C until time of analysis. Analysis is to be performed within 28 days of sampling.

7.2 Soils/sediment samples

7.2.1 Samples should be collected in glass jars and stored in a field-moist condition. Ammonia may be lost by volatilization due to drying or thawing.

7.3 Extraction is to be performed within 7 days of sampling. Extracts may be preserved with 9N sulfuric acid and stored for up to 28 days until analysis.

8) Standards, Reagents, and Consumable Materials

8.1 Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking (ADM-RLT)* for the complete procedure and documentation requirements.

8.2 All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I or ASTM Type II reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

8.3 Reagents and Standards must comply with the traceability, labeling and documentation practices specified in the SOPs: *Making Entries onto Analytical Records* and *Reagent and Standards Login and Tracking* (ADM-RLT).

8.1 Dechlorinating reagents

8.1.1 Sodium thiosulfate: Dissolve 3.5 g $Na_2S_2O_3C5{\cdot}H_2O$ in reagent water and dilute to 1L. One mL of this solution will remove 1mg/L of residual chlorine in 500mL of sample.

8.1.2 Sodium sulfite: Dissolve 0.9g Na_2SO_3 in reagent water and dilute to 1L. One mL removes 1mg/L Cl per 500mL of sample.

8.2 Sodium Hydroxide Solution, 6N, dissolve 48g of NaOH pellets in 150mL deionized water. Dilute to 200mL, cool and store in plastic container.

8.3 KCl, 2M, dissolve 150g of KCl in 1L deionized water.

8.4 Boric Acid Solution, 2% (w/v), dissolve 20.0g of H3BO3 in deionized water. Dilute to final volume of 1,000mL.

8.5 Sulfuric acid, 0.04N, 1.1mL of concentrated sulfuric acid to 1L DI water.

8.6 Sulfuric acid, 9N.

8.7 Bran & Luebbe analyses - For analysis performed on the Bran & Luebbe analyzer, refer to the Bran & Luebbe method attachment for reagents used and preparation instructions.

8.7.1 Stock Standard 10,000mg/L: In a 1L volumetric flask containing approximately 800mL of DIW dissolve 38.19g of ammonium chloride (NH_4Cl) that has been dried for two hours at 110° C. Dilute to the mark and invert to mix. The stock standard is stored at room temperature and is good for six months from the date of preparation.

8.7.2 Working Standard 100mg/L: To a 100mL volumetric flask containing DIW add 1mL of 10,000ppm stock standard. Dilute to the 100mL mark. The working standard is stored at room temperature and is good for seven days from the date of preparation.

8.7.3 Prepare a series of at least three standards and a blank by diluting a known amount into 100mL volumetric flask. See Table 1 and Table 2 for preparation amounts and concentrations. The calibration standards are stored at room temperature and are good for seven days from the date of preparation.

NOTE: Standards for soil extracts are prepared as in Table 1, except that the standards are diluted into **2M KCL INSTEAD of DIW.**

8.7.4 Since the intensity of the color used to quantify the concentration is pH dependent, the acid concentration of the wash water and the standard ammonia solutions should approximate that of the samples.

8.8 Skalar Analyzer

8.8.1 Buffer Solution A (1 Liter)

8.8.1.1 di Sodium EDTA 3g

8.8.1.2 C10H14N2Na2O8.2H2O

8.8.1.3 Boric acid 3g

8.8.1.4 H_3BO_3

8.8.1.5 NaOH 40g

8.8.1.6 Distilled water H_2O

8.8.1.7 Brij 35 (30%) 2mL

8.8.1.8 Dissolve the sodium hydroxide, di-sodium EDTA and boric acid in ±800mL distilled water. Fill up to 1L with distilled water; add the Brij 35 and mix. Degas the solution by filtration.

8.8.1.9 The pH of the solution should be 12-13. The solution is stable for 1 month when stored in a polyethylene bottle at room temperature.

8.8.2 Buffer solution B (1L)

8.8.2.1 Potassium sodium tartrate 33g

8.8.2.2 $C_4H_4O_6KNa.4H2O$ - tri-Sodium citrate 24g

8.8.2.3 Tri-Sodium citrate 24g

8.8.2.4 $C_6H_5O_7Na_3.2H_2O$

8.8.2.5 Distilled water

8.8.2.6 Brij 35 (30%) 2mL

8.8.2.7 Dissolve the potassium sodium tartrate in ±800mL distilled water. Add the tri- sodium citrate and dissolve. Fill up to 1L with distilled water, add the Brij 35 and mix.

8.8.2.8 Check the pH and adjust if necessary with hydrochloric acid to 5.2±0.1. Solution is stable for 1 week. Store at 4˚C when the solution is not used.

8.8.3 Solution C: Sodium salicylate solution (1L)

8.8.3.1 Sodium hydroxide 25g.

8.8.3.2 Sodium salicylate 80g C_7H_5NaO3

8.8.3.3 Distilled water H_2O

8.8.3.4 Dissolve the sodium hydroxide in ±50mL distilled water. Add ±800mL distilled water. Add the sodium salicylate. Fill up to 1L with distilled water and mix.

8.8.3.5 Store in a dark colored bottle. Solution is stable for one week. Store at 4˚C when the solution is not used.

8.8.4 Solution D Sodium nitroprusside solution (1L)

8.8.4.1 Sodium nitroprusside 1.0g

8.8.4.2 Na_2 $[Fe(CN)5NO].2H_2O$

8.8.4.3 Distilled water H_2O

8.8.4.4 Dissolve the sodium nitroprusside in ±800mL distilled water. Fill up to 1L with distilled water and mix.

8.8.4.5 Store in a dark colored bottle. Solution is stable for one week. Store at 4˚C when the solution is not used.

8.8.5 Solution E: Sodium dichlororisocyanurate solution (1L)

8.8.5.1 Sodium dichlororisocyanurate 1.0g $C_3N_3O_3Cl_2Na.2H_2O$

8.8.5.2 Distilled Water

8.8.5.3 Dissolve the sodium dichlorisocyanurate in ±800mL distilled water. Fill up to 1L with distilled water and mix.

8.8.5.4 Solution is stable for 1 week. Store at 4˚C when the solution is not used.

8.8.5.5 In case the calibration is 2nd order (hollow), the amount of dichlororisocyanurate can be reduced until the calibration is linear.

8.8.6 Solution F. Rinsing liquid sampler (ammonia, water no. 1.2.3) (1L)

8.8.6.1 Sulfuric acid 2mL H_2SO_4 (95-97%)

8.8.6.2 Distilled Water

8.8.6.3 Add the sulfuric acid to the distilled water until the pH <2, and mix.

8.8.6.4 Solution is stable for one week.

8.8.7 Solution G: Rinsing liquid sampler (ammonia soil extract no. 2.2.8X 2M potassium chloride solution) (1L).

8.8.7.1 Potassium chloride 149g

8.8.7.2 KCl

8.8.7.3 Distilled water H_2O

8.8.7.4 Dissolve the potassium chloride in ±800mL distilled water. Fill up to 1L and mix.

9) Apparatus and Equipment

9.1 Flow Injection Analyzer with associated software and computer system. The two systems currently in use are listed below.

9.1.1 Bran & Luebbe Auto Analyzer

9.1.2 Skalar Segmented Flow Analyzer

9.2 Heating unit associated with the FIA

9.3 Wrist Action Shaker

9.4 Filter Funnel and Whatman 934-AH or equivalent

9.5 Glassware

9.6 Standard laboratory glassware

9.7 Class-A volumetric flasks and pipettes

10) Preventative Maintenance

10.1 All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described herein. The entry in the log must include: date of event, the initials of who performed the work, corrective action and a reference to analytical control.

10.2 Change reagent and sampling lines when they are flat.

10.3 For problems encountered with the analyzer operation, refer to the applicable manufacturer's operating and/or troubleshooting information.

11) Procedure

11.1 Sample preparation – water samples

11.1.1 All samples must be distilled unless otherwise required or specified by

the client.

11.2 Soil sample extraction

11.2.1 Weigh out 10g of sample into a 50mL centrifuge tube.

11.2.2 Spike appropriate QC sample with 0.5mL of 10,000ppm NH_3 stock solution.

11.2.3 Bring to 50mL volume with 2M KCl. The Method blank contains only KCl.

11.2.4 Shake samples for 30 minutes on the wrist-action shaker.

11.2.5 Centrifuge samples for 10-20 minutes.

11.2.6 Filter supernatant with 0.45µm filter. Samples are now ready for analysis.

11.3 Soil and sediment extraction – Plumb extraction procedure:

11.3.1 Weigh 20g of sample into a 50mL centrifuge tube.

11.3.2 Spike appropriate QC sample with 1.0mL of 10,000ppm NH_3 stock solution.

11.3.3 Bring to 50mL volume with 2M KCl. The Method blank contains only KCl.

11.3.4 Shake samples for 30 minutes on the wrist-action shaker.

11.3.5 Centrifuge samples for 10-20 minutes.

11.3.6 Filter supernatant with 0.45µm filter into a 100mL volumetric flask.

11.3.7 Wash the remaining solids with an additional 50mL of 2M KCl and filter into the volumetric flask. Bring to volume with 2M KCl. Samples are now ready for analysis.

11.4 Calibration and Sample Analysis – Bran & Luebbe System

11.4.1 Set up instrument according to the Bran & Luebbe method US-696D-82X (See Attachments).

NOTE: 2M KCl may be used for a sample carrier when running extracts instead of DIW.

11.4.2 Analyze the calibration standards listed in Table 1 (regular level) or Table 2 (low level). The calibration curve is fitted to a linear, least squares model. See Table 4. The correlation coefficient must be >0.995.

11.4.3 After the calibration is established, it must be verified by the analysis of an ICV or a LCS if prepared from an independent source. If the ICV is within ±10% of the true value, then analysis may proceed (see Table 3). If not, determine the cause of the problem, correct it, and then recalibrate.

11.4.4 Ensure that the pH of the dilution water and standard NH_3 solution approximates that of the samples. Place samples and QC aliquots in sampler vials and arrange on autosampler according to Table 3.

11.4.5 Repeat sequence bracketing every 10 samples with CCV and CCB. End the sample tray (sequence) with CCV and CCB.

11.5 Calibration and Sample Analysis- Skalar Analyzer

11.5.1 Set up instrument according to the Skalar method Catnr. 156-350.1w/rX(+P3) issue 011917/99315243 See Section 19.8.

11.5.2 Prepare the working standards fresh daily. The water used for the reagents and standards must be degassed properly before making up the reagents and standards. Especially water produced by reverse osmosis or ion exchange equipment contains a lot of gasses, which must be removed by degassing (degassing procedure see Operational remarks and troubleshooting).

11.5.3 Analyze the calibration standards listed in Table 5. The calibration type is 1st order ISO 8466-1. The calibration curve is fitted to a linear, least squares model. See Appendix A. The correlation coefficient must be > 0.995.

11.5.4 After the calibration is established, it must be verified by the analysis of an ICV or a LCS if prepared from an independent source. If the ICV is within ±10% of the true value, then analysis may proceed (see Table 4). If not, determine the cause of the problem, correct it, and then recalibrate.

11.5.5 Ensure that the pH of the dilution water and standard NH3 solution approximates that of the samples. Place samples and QC aliquots in sampler vials and arrange on autosampler according to Table 3. Repeat sequence bracketing every 10 samples with CCV and CCB. End the sample tray (sequence) with CCV and CCB.

12) QA/QC Requirements

12.1 This method shall operate under the formal Quality Assurance Program established at ALS and must maintain records that define the quality of data that is generated. Data shall be compared to established criteria in order to determine if the results of the analyses meet the performance characteristics of the method. It is required that an initial demonstration of capability and periodic analysis of laboratory reagent blanks, laboratory fortified blanks, and other QC solutions as a continuing check on performance. The accuracy and precision of the procedure must be validated *before* analyses of samples begin, or whenever significant changes to the procedures have been made.

12.2 Initial Precision and Recovery Validation

12.2.1 The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed. Performance is acceptable if the % recovery and % RSD meet LCS (or method) acceptance limits.

12.3 Method Detection Limits and Method Reporting Limits

12.3.1 A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike seven blank matrix (water or soil) samples with MDL spiking solution at a level below the MRL. Follow the analysis procedures in to analyze the samples.

12.3.2 Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.* The MDL study must be verified annually.

12.3.3 Limits of Quantification (LOQ)

12.3.3.1 The laboratory establishes a LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard or extract prepared at the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries should be within 50-150% of the true values to verify the data reporting limit. Refer to Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.

12.4 Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management (ADM-DOD).* General QC Samples are:

12.4.1 The Linear Calibration Range (LCR) must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve falls in the linear range of the instrument. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ±10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

12.4.2 CCV (Continuing Calibration Verification): A CCV is analyzed every 10 samples using 2.0mg/L NH_3 standard (from curve). Recovery must fall within 90-110% or 1.8-2.2mg/L.

12.4.3 CCB (Continuing Calibration Blank): A blank consisting of the carrier solution (DIW or KCl). The result must be less than the absolute value of the Method Reporting Limit - $|MRL|$.

12.4.4 Method Blank

12.4.4.1 A method blank is extracted and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis. For some project specific needs, exceptions may be noted and method blank results above the

MRL may be reported for common lab contaminants (phthalate esters, etc.).

12.4.5 Lab Control Sample (LCS)

12.4.5.1 The laboratory control sample is composed of analyte-free water or solid matrix (sodium sulfate or sand) into which is spiked a number of appropriate target analytes. The LCS is designed to monitor the accuracy of the procedure. The concentration of the spike in the LCS matrix should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

12.4.5.2 A lab control sample (LCS) must be prepared and analyzed with every batch of 20 (or fewer) samples. Calculate the LCS recovery as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

12.4.5.3 The acceptance criteria are given in Table 4. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and corrective action is taken. The samples are re-extracted or re-analyzed or the data reported with the appropriate qualifiers.

12.4.6 Matrix Spike

12.4.6.1 A matrix spike (MS) and duplicate matrix spike (DMS) must be prepared and analyzed with every batch of 10 (or fewer) samples. The MS/DMS is prepared by adding a known volume of the matrix spike solution to the sample and determining the spiked sample concentration. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

12.4.6.2 Calculate Relative Percent Difference (RPD) as:

$$\%RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where R1= Higher Result
R2= Lower Result

12.4.6.3 Calculate Relative Standard Deviation (RSD) as:

$$\%RSD = \frac{stddev}{mean} x\,100$$

12.4.6.4 The acceptance range for %RPD or %RSD is ≤20%. If the RPD is within the acceptance range, the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.

12.4.6.5 Following analysis of the MS the percent recovery is calculated and compared to acceptance limits given in the Table 4. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

13) Data Reduction and Reporting

13.1 Data Reduction

13.1.1 At the end of the analysis sequence, the calibration data is printed including the calculated standards concentrations, calibration statistics, and slope and intercept of the calibration curve.

13.1.2 The runtime report printed during the sampling run is turned in with a final report and calibration report.

13.1.3 Raw results with corresponding dilution factors are uploaded to an analyst-created run in LIMS. LIMS then calculates the final reported values.

13.2 Reporting

13.2.1 Refer to the SOP for *Data Reporting and Report Generation* for reporting guidelines.

13.2.2 Reports are generated in the ALS LIMS by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transferred to a file which LIMS uses to generate a report. The forms generated may be ALS standard reports, DoD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.2.3 Report results to three significant figures. If sample was diluted report two significant figures.

13.2.4 Soil and sediment samples should be reported as method 350.1 Modified or SM 4500-NH3 G Modified.

13.3 Data Review and Assessment

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process (ADM-DREV)* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Manager to inclusion in the report narrative.

13.3.1 Bench sheets are completed and a batch lot number is assigned. The Manufacturer's lot numbers or ID's for the reagents are added to bench sheets.

14) Contingencies for Handling Out-of-Control or Unacceptable Data

14.1 Refer to the SOP for *Nonconformity and Corrective Action* (ADM-NCAR) for procedures to records and the proper actions for out of control events.

15) Method Performance

15.1 The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. The method detection limit (MDL) is established using the procedure described in the SOP *Performing and Documenting Method Detection Limit Studies and Estimation of Limits of Detection and Quantitation.*

16) Pollution Prevention and Waste Management

16.1 It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

16.2 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

16.3 This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS Kelso Chemical Hygiene Plan for details.

16.4 This method uses a base. Waste base is hazardous to the sewer system and to the environment. All waste must be neutralized prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be

documented on the treatment by generator record. See the ALS Kelso Chemical Hygiene Plan for details.

17) Training

17.1 All analysts performing this analysis are required to read and understand this SOP.

17.2 Training is documented following the *Employee Training and New Employee Orientation* (ADM-TRAIN).

18) Method Modifications

18.1 The reference method is modified to allow for the analysis of solid or semi-solids.

19) References

19.1 TNI Standard, Volume, 2009; 2016.

19.2 ISO/IEC 17025:2005, 2017.

19.3 DoD Quality Systems Manual for Environmental Laboratories, current version.

19.4 Method 350.1 Determination of Ammonia Nitrogen by Semi-Automated Colorimetry, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Chemistry Research Division, Revision 2.0, August 1993.

19.4.1 G:\QA Controlled Documents\Manuals_QAPPs\350.1 Instrument manuals and methods\350.1.pdf.

19.5 SM 4500-NH3 G-2017, Standard Methods for the Examination of Water and Wastewater.

19.6 Skalar SFA SA5000.

19.6.1 G:\QA Controlled Documents\Manuals_QAPPs\Skalar\Methods\ammonia & total nitrogen (gas dialysis) skalar.pdf

19.7 Bran & Luebbe Method US-696D-82X.

19.7.1 G:\QA Controlled Documents\Lab SOPs\GEN\350.1 Instrument manuals and methods\350.1 B&L.pdf.

19.8 Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound, January, 1996.

19.9 Procedures for Handling and Chemical Analysis of Sediment and Water Samples, Russell S. Plumb, U.S. EPA/Corps of Engineers Technical Committee on Criteria for Dredged Fill Material, May 1981.

20) Changes since Last Revision

Revision Number	Effective Date	Document Editor	Description of Changes
15.0	5/27/2020	T.Caron	Reformatted SOP to current ALS branding. Minor typographical, grammatical, and formatting revisions to improve readability and consistency. Administrative changes at scheduled review cycle.

16.0	4/26/2022	E. Davelaar	Updated SOP Signatories. Removed references to Environmental Express SimpleDist system and Lachat analyzer throughout SOP. These are no longer used. Section 1.1: Updated aqueous samples, updated SM 4500 NH3 G-2017 from G-2011. Section 2.1: Updated in its entirety. Section 2.2: Updated in its entirety. Section 13.1.3: Updated in its entirety. Section 13.2.4: Removed reference of undistilled water samples and added in reporting of SM 4500-NH3 G Modified. Section 19.5: Updated to SM 4500-NH3 G-2017. Inserted Section 19.6.

21) Attachments, Tables, and Appendices

21.1 Table 1: Calibration Standards Preparation, Regular Level Ammonia.

21.2 Table 2: Calibration Standards Preparation, Low Level Ammonia.

21.3 Table 3: Analysis Run Scheme.

21.4 Table 4: Summary of Corrective Actions.

21.5 Table 5: Routine Calibration Standards Preparation SKALAR METHOD.

TABLE 1
Calibration Standards Preparation
Regular Level Ammonia

Routine Calibration Standards

Volume of 100 µg/ml Solution (ml)	Final Volume (ml)	Standard Concentration (mg/L)
0	100	0.00
0.05	100	0.05
0.50	100	0.50
2.00	100	2.00
5.00	100	5.00

TABLE 2
Calibration Standards Preparation
Low Level Ammonia

Low Level Calibration Standards

Volume of 10 µg/ml Solution (ml)	Final Volume (ml)	Standard Concentration (mg/L)
0	100	0.00
0.10	100	0.01
0.50	100	0.05
2.50	100	0.25
5.00	100	0.50

Note: For low level analysis, change sample tubing from 0.16”ID (orn/yel) to 0.42”ID (orn/orn).

TABLE 3
ANALYTICAL RUN SCHEME

STEP	SAMPLE
1	ICV
2	CCB
3	CCV-1
4	CCB-1
5	MB
6	LCS
7	SAMPLE
8	SAMPLE DUP
9	SAMPLE SPIKE
10	SAMPLE SPIKE DUP
11	SAMPLE
12	SAMPLE
13	SAMPLE
14	SAMPLE
15	CCV-2
16	CCB-2
17-26	CCB-3 REPEAT STEPS 5-28 FOR REMAINDER OF SAMPLES.

TABLE 4

Summary of Corrective Actions

Method Reference	Control	Specification and Frequency	Acceptance Criteria	Corrective Action
EPA 350.1, 350.1M SM 4500-NH3 G-2011	ICAL	Prior to sample analysis	R2 ≥ 0.995	Correct problem then repeat ICAL
EPA 350.1 350.1M SM 4500-NH3 G-2011	ICV	After ICAL	± 10%	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
EPA 350.1 350.1M SM 4500-NH3 G-2011	CCV	Prior to sample analysis	± 10%	Correct problem then repeat CCV or repeat ICAL
EPA 350.1 350.1M SM 4500-NH3 G-2011	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20x amount in blank.
EPA 350.1 350.1M SM 4500-NH3 G-2011	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	90-110	If exceeds limits, re-extract and re-analyze
EPA 350.1 SM 4500-NH3 G-2011 350.1M	Matrix Spike	Include per 10 samples – 350.1; Per 20 samples – SM 4500-G	90-110 55-135mg/kg	Evaluate data to determine if the there is a matrix effect or analytical error.
EPA 350.1 SM 4500-NH3 G -2011	Sample Duplicates	Include with each analysis batch (up to 20 samples)	20	Re-homogenize and re-analyze if result is > 5 X the MRL

TABLE 5

Routine Calibration Standards Preparation
SKALAR Method - Ammonia

Volume of 100 µg/ml Solution (ml)	Final Volume (ml)	Standard Concentration (mg/L)
0	100	0.00
0.05	100	0.05
0.10	100	0.10
0.50	100	0.50
1.00	100	1.00
2.00	100	2.00

NITRATE/NITRITE, NITRITE BY AUTOMATED COLORIMETRY

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1) Scope & Applicability

1.1 The purpose of this procedure is to determine the concentration of Nitrite, or Nitrate/Nitrite in water, wastewater and soil samples using EPA Method 353.2. This procedure is applicable to determination of Nitrite or Nitrate/Nitrite concentrations greater than 0.02mg/L in water, wastewater, and leachates from solids. The Method Reporting Limits (MRLs) and Method Detection Limits (MDLs) are listed in the ALS Kelso DQO Table and Table 1.

1.2 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management,* may supersede the requirements defined in this SOP.

2) Summary of Procedure

2.1 A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite is then determined by diazotization with sulfanilamide, and coupling with N-(1-napthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically. Separate, rather than combined nitrate/nitrite, values are obtained by carrying out the procedure first with, and then without the Cd reduction column, and subtracting NO_2 from NO_3/NO_2 to obtain NO_3.

2.2 Soil samples may be analyzed following an extraction and filtration procedure and is cited as 353.2M.

3) Definitions

3.1 For laboratory definitions applicable to most analyses, refer to the SOP for *Sample Batches*.

4) Responsibilities

4.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. The department supervisor/manager or designee performs final review and sign-off of the data.

5) Interferences

5.1 Suspended solids can clog the reduction column and restrict sample flow. Since nitrate/nitrite is found in a dissolved state, the sample may be filtered before analysis.

5.2 Dissolved metals may cause low results. EDTA can be added to the samples to eliminate this interference. Also, 10N NaOH can be added to the samples to

precipitate the metals and eliminate this interference.

5.3 The buffer in this method allows analysis of sulfuric acid-preserved samples without pH adjustment. Negative peaks during flow injection analysis, or peaks which split into groups, are an indication that samples have been excessively acidified, and must be adjusted to a pH between 5.0 and 8.0 with 10N NaOH or NH_4OH prior to analysis.

5.4 Samples which contain large amounts of oil and grease will coat the surface of the cadmium. The sample may be pre-extracted with an organic solvent to alleviate this interference.

5.5 Flow Injection Analysis (FIA) – Highly colored samples may require background correction. To perform a background correction, run the samples through the flow injection analyses without the sulfanilamide reagent. Run buffer through the sulfanilamide line in place of the color reagent.

6) Safety

6.1 Chemicals, reagents and standards must be handled as described in the ALS Kelso safety policies, approved methods and in SDSs where available. Refer to the ALS Kelso Environmental, Health and Safety Manual and the appropriate SDS prior to beginning this method.

6.2 Sodium Hydroxide (NaOH) is a strong caustic and a severe health and contact hazard. Use nitrile or latex gloves while handling pellets or preparing solutions.

6.3 Be cautious of fumes when preparing the NH_4Cl buffer.

6.4 Dispose of culture tubes in the broken glass container.

6.5 Empty waste containers in the hood.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Aqueous samples for nitrate+nitrite are collected in plastic containers and must be preserved with H_2SO_4 to a pH of 2 or less, and analyzed within 28 days from collection.

7.2 If nitrate and nitrite are to be determined separately, the sample is not preserved, must be stored at ≤6ºC, and analyzed within 48 hours from collection.

7.3 Soil samples are collected in 4oz. glass or plastic containers and stored at 4±2ºC. Extraction is performed within 7 days of sampling. Extracts are preserved with 9N H_2SO_4 and stored up to 28 days until analysis.

8) Standards, Reagents, and Consumable Materials

8.1 Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP Reagent/Standards Login and Tracking for the complete procedure and documentation requirements.

8.2 All stocks, working solutions and sample dilutions should be prepared using

deionized water (DI) conforming to ASTM Type I or ASTM Type II reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

8.3 10N NaOH: Add 100g NaOH to 250mL DI water very slowly, and swirl to dissolve (reaction is highly exothermic).

8.4 Ammonium Chloride Buffer (Lachat): In a hood, to a 1L volumetric flask, add 500mL DI water, 105mL HCL, 95mL NH4OH and 1.0g disodium EDTA. Stir until dissolved. Adjust the pH to 8.5 with HCL or 15N NaOH.

8.5 Sulfanilamide Color Reagent (Lachat): Add 600mL of DI water and 100mL phosphoric acid to a 1000mL volumetric flask. Then add 40.0g sulfanilamide and 1.0g N-(1-naphthyl) ethylene diamine dihydrochloride to the flask, and shake to wet. Mix with a magnetic stirrer until all dry chemical is dissolved (approximately 30 minutes), remove the stir bar, and dilute to the mark with DI water. Store the reagent in a labeled brown glass bottle for no more than one month.

8.6 Copper Sulfate Stock Solution: Dissolve 2.5g copper sulfate in 600mL DI water. Dilute to one liter and mix thoroughly.

8.7 Nitrate Stock Solution: In a 100mL flask, dissolve 6.068g dried $NaNO_3$ to 100mL final volume with DIW. Concentration =10,000ppm A Nitrate Standard shall be used for the ICV. Solution is stable for six months.

8.8 Nitrite Stock Solution: In a 250mL flask, dissolve 12.316g dried $NaNO_2$ to 250mL final volume with DIW. Concentration =10,000ppm. It is standard practice to use the Ion Chromatography Stock. Solution is stable for six months.

8.9 Working Nitrate Solution: Dilute 1.0mL of Stock Nitrate Solution to 100mL with DI water. 1mL=100µg NO_3-N. Prepare fresh weekly.

8.10 Working Nitrite Solution: Dilute 1.0mL of Stock Nitrite Solution to 100mL with DI water. 1mL=100µg NO_2-N. Prepare fresh weekly.

8.11 Calibration Standards: Prepare a series of standards according to one of the options in the Calibration Tables (Reference Appendix). All standards are prepared with DI water.

8.12 KCl, 2M: dissolve 149.1g of KCl in 1L deionized water.

8.13 2% Copper Sulfate Solution: Dissolve 10g of copper sulfate pentahydrate $CuSO_4 \cdot 5H_2O$ in approximately 400mL of reagent water and dilute to 500mL.

8.14 2N Hydrochloric Acid: Cautiously, with stirring, slowly add 83.0mL of concentrated hydrochloric acid HCl to approximately 300mL of reagent water. Cool to room temperature and dilute to 500mL with reagent water.

8.15 6N Hydrochloric Acid: Cautiously, with stirring, slowly add 50mL of concentrated hydrochloric acid HCl to approximately 30mL of reagent water. Cool to room temperature and dilute to 100mL with reagent water.

8.16 2N Nitric Acid: Cautiously, with stirring, slowly add 12.8mL of concentrated nitric acid HNO_3 to approximately 60mL of reagent water. Cool to room temperature and dilute to 100mL with reagent water.

9) Apparatus and Equipment

9.1 Lachat QuikChem AE Flow Injection Analyzer and Data System.

9.2 Copper-Cadmium Reduction Column - Lachat Part #50237. For WestCo SmartChem Discrete Analyzer use open tubular copperized cadmium redactor (OTCR) Part #165-0301-01.

9.3 Standard laboratory glassware and digital pipettors for preparing reagents and dilutions.

9.4 Wrist action shaker.

10) Preventative Maintenance

10.1 All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described herein. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

10.2 Lachat

10.2.1 Change reagent and sampling lines when they are flat for the FIA instruments.

10.2.2 If problems encountered with operations for the Lachat see the instruments troubleshooting guide for guidance

10.3 Cleaning of the hydraulic line and cuvette can be performed more rigorously when discoloration of these parts is apparent. Refer to the Westco Scientific Instruments, Inc. Maintenance Manual, Chapter 7.7 for this procedure.

11) Procedure

11.1 Calibration and Standardization - Flow Injection Analysis

11.1.1 Prepare a series of at least three standards, covering the desired range (Ref. Table 3), and a blank by diluting suitable volumes of standard nitrate solution. At least one nitrite standard should be compared to a nitrate standard of same concentration to verify efficiency of the reduction column.

11.1.2 Place appropriate standards in the sampler in order of decreasing concentration and perform analysis.

11.1.3 Prepare standard curve by plotting instrument response against concentration values. Use linear regression to establish calibration curve against concentration/response data. An acceptable linear curve should have a linear coefficient (r value) of 0.995 or better.

11.1.4 After calibration has been established, it must be verified by the analysis of a suitable quality control sample, Initial Calibration Verification (ICV), which is independent from the stock solution used for calibration. If measurements exceed ±10% of the true ICV value, terminate the analysis and recalibrate the instrument.

11.2 Sample Preparation

11.2.1 Water samples do not require a preparation step. Filtering is included in analysis procedures below.

11.2.2 For soil sample extraction, perform the following steps.

11.2.2.1 Weigh out 10g of sample into a 50mL centrifuge tube.

11.2.2.2 Spike appropriate QC sample with 0.5mL of 10,000ppm NO_3 stock solution.

11.2.2.3 Bring to 50mL volume with 2M KCl. The Method blank contains only KCl.

11.2.2.4 Shake samples for 30 minutes on the wrist-action shaker.

11.2.2.5 Centrifuge samples for 10-20 minutes.

11.2.2.6 Filter supernatant with 0.45µm filter. Samples are now ready for analysis.

11.3 Procedure for Lachat QUIKCHEM AE Analyzer

11.3.1 Turn both the Lachat and computer on. Let warm up for ~20 minutes.

11.3.2 De-gassing of carrier and buffer with helium for 5 minutes is recommended if baseline spikes occur.

11.3.3 Load method and set up instrument according to Lachat QuikChem AE method.

11.3.4 Fasten reagent lines across the pump and into the reagent bottles. Start pump.

11.3.5 Calibrate with nitrate standards appropriate to the expected NO_3/NO_2 levels in the sample.

11.3.6 Connect cadmium column at the 2nd valve so the sample runs through the column before coloring. For $NO_3 \rightarrow NO_2$ reduction.

11.3.7 For NO_2 determination calibrate with NO_2 standards.

11.3.8 Filter samples through a 0.45µm syringe type membrane filter into auto sampler vials, and arrange the run according to Table 2.

11.3.9 Start calibration and sample tray after reagents flow through the flow cell and a steady baseline appears. Refer to Table 3 for a typical run sequence. A new curve must be prepared every six months or whenever the CCV fails, assuming no significant changes to the instrument or to the method has been made.

11.3.10 CCV (Continuing Calibration Verification) – A CCV must be analyzed following every tenth vial on the autosampler. The CCV is a 1.0mg/L NO_3-N standard and the recovery must be ±10% of the expected value. The CCV for NO_2-N only is a 1.0mg/L NO_2 standard.

11.3.11 CCB (Continuing Calibration Blank) – A CCB must be analyzed following every CCV. The CCB is a portion of DI water, and the result must be below the MRL.

11.3.12 Cleanup – Remove the cadmium column. Pump 1N HCl across the board lines followed by DI water. Pump air through the lines to dry.

12) QA/QC Requirements

12.1 This method shall operate under the formal Quality Assurance Program established at ALS and must maintain records that define the quality of data that is generated. Data shall be compared to established criteria in order to determine if the results of the analyses meet the performance characteristics of the method. It is required that an initial demonstration of capability and periodic analysis of laboratory reagent blanks, laboratory fortified blanks, and other QC solutions as a continuing check on performance. The accuracy and precision of the procedure must be validated *before* analyses of samples begin, or whenever significant changes to the procedures have been made.

12.2 Linear Calibration Range (LCR)

12.2.1 The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ±10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

12.3 Method Detection Limits and Method Reporting Limits

12.3.1 A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike seven blank matrix (water or soil) samples with MDL spiking solution at a level below the MRL. Follow the analysis procedures in to analyze the samples.

12.3.2 Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.* The MDL study must be verified annually.

12.3.3 Limits of Quantification (LOQ)

12.3.3.1 The laboratory establishes a LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard or extract prepared at the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries should be within 50-150% of the true values to verify the data reporting limit. Refer *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.*

12.3.4 The Method Reporting Limits (MRLs) used at ALS are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which ALS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

12.4 Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management.* General QC Samples are:

12.4.1 Method Blank

12.4.1.1 A method blank is extracted and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants.

12.4.2 Lab Control Sample (LCS)

12.4.2.1 The laboratory control sample is composed of analyte-free water or solid matrix (sodium sulfate or sand) into which is spiked a number of appropriate target analytes. The LCS is designed to monitor the accuracy of the procedure. The concentration of the spike in the LCS matrix should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

12.4.2.2 A lab control sample (LCS) must be prepared and analyzed with every batch of 20 (or fewer) samples. Calculate the LCS recovery as follows:

$$\%R = X/TV \times 100$$

Where: X = Concentration of the analyte recovered
TV = True value of amount spiked

12.4.2.3 The acceptance criteria are given in the ALS Kelso DQO Table. If the LCS fails acceptance criteria, corrective action must be taken. Corrective action includes recalculation, reanalysis.

12.4.3 Matrix Spike

12.4.3.1 A matrix spike (MS) and duplicate matrix spike (DMS) must be prepared and analyzed with every batch of 10 (or fewer) samples. The MS/DMS is prepared by adding a known volume of the matrix spike solution to the sample and determining

the spiked sample concentration. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

12.4.3.2 Calculate Relative Percent Difference (RPD) as:

$$\%\,RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where R1= Higher Result
R2= Lower Result

12.4.3.3 Following analysis of the MS the percent recovery is calculated and compared to acceptance limits in the ALS Kelso DQO Table.

12.4.3.4 If the MS recovery is within control limits the results are reported.

12.4.3.5 If the MS recovery is not within control limits, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. Verify that the spike level is at least 5X the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

12.4.4 Sample Duplicates - One sample per batch of 10 or fewer samples must be analyzed in duplicate. Relative Percent Difference must be $\leq$20%.

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where R1= result for the sample
R2= result for the sample duplicate

Prior to preparation of samples, blanks should be analyzed to determine possible interferences from sample handling steps, reagents, or glassware. If the blanks show contamination, the source of the contamination should be isolated and minimized.

13) Data Reduction and Reporting

13.1 Calculations

13.1.1 For water samples, the instruments will readout the sample concentration values. No additional calculations are needed other than taking into account any dilutions made.

13.1.2 For soil samples, the instruments will readout the analyzed sample concentration. These are used to calculate the soil sample concentration, taking into account the initial weight, final volume, and any dilutions made.

13.2 Data Reporting

13.2.1 Refer to the SOP for *Laboratory Data Review Process* for general instructions for data review.

13.2.2 Report results as follows:

13.2.2.1 For Nitrate/Nitrite as "Nitrate/Nitrite as Nitrogen" in mg/L units.

13.2.2.2 Report results for Nitrite as "Nitrite as Nitrogen" in mg/L units.

13.2.2.3 Report results for Nitrate as "Nitrate as Nitrogen" in mg/L units.

13.2.2.4 Soil samples should be reported as method 353.2 Modified in mg/kg (dry wt. basis).

13.2.2.5 Report results to 3 significant figures unless the sample was diluted. If a dilution was performed, report 2 significant figures.

13.2.2.6 It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in this SOP. Average, RPD, spike level and spike recovery are entered on the analytical spreadsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

13.2.2.7 Reports are generated in the ALS LIMS by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transferred to a file which Excel© uses to generate a report. The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.3 Data Review and Assessment

13.3.1 Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the

report is also reviewed. Refer to the *SOP for Laboratory Data Review Process (ADM-DREV)* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Chemist to inclusion in the report narrative.

13.3.2 It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in this SOP. Average, RPD, spike level and spike recovery are entered on the analytical spreadsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

14) Method Performance

14.1 The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS aliquots are prepared and analyzed. The average percent recovery must meet the laboratory control sample acceptance limits.

15) Pollution Prevention and Waste Management

15.1 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

15.2 This method uses a base. Waste base is hazardous to the sewer system and to the environment. All waste must be neutralized prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS Lab Waste Management Plan for details.

16) Contingencies for Handling Out-of-Control or Unacceptable Data

16.1 Refer to the SOP for *Nonconformity and Corrective Action* (ADM-NCAR) for corrective action procedures and to document the proper actions for out of control events.

16.2 Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

17) Training

17.1 All analysts performing this analysis are required to read and understand this SOP.

17.2 Training is documented following the *Employee Training and New Employee Orientation* (ADM-TRAIN).

17.3 It is required that an initial demonstration of capability and periodic analysis of laboratory reagent blanks, laboratory fortified blanks, and other QC solutions as a continuing check on performance.

18) Method Modifications

18.1 Soil samples may be analyzed following an extraction and filtration procedure and is cited as 353.2M.

19) References

19.1 Standard Methods for the Examination of Water and Wastewater, 22nd Ed.

19.2 SEPA, *Methods for Chemical Analyses of Water and Wastes,* EPA-600/4-79-020, August 1993 - Method 353.2 Revision 2.0.

19.3 Lachat QuikChem Methods.

19.4 TNI Standard, Volume 1- 2009, TNI Standard, Volume 1 -2016.

19.5 DoD Quality Systems Manual for Environmental Laboratories Current Version.

19.6 ISO/17025:2017 American National Standard, General Requirements for the Competence of Testing and Calibration Laboratories.

20) Changes Since Last Revision

Revision Number	SOP Review	Document Editor	Description of Changes
12.0 12.0	 J. Coronado 2.23.2021	T. Caron	Admin Changes only not affecting technical content. Documented date of annual SOP Review, updated SOP signatories; boiler plate standard paragraphs have been updated to reflect current practices. Reviewed and approved; no technical changes at this time.
13.0		E. Davelaar	Updated SOP Signatories. Section 11.3.2: Updated de-gassing if baseline spikes occur. Section 11.3.8: Deleted adjusting of pH. Section 11.3.12: Removed first step of cleanup.

21) Attachments, Tables, and Appendices

21.1 Table1: Analytical Run Scheme – Lachat.

21.2 Table 2: Summary of Corrective Actions.

TABLE 1
Analytical Run Scheme
Lachat

Step	Sample
1	ICB
2	ICV
3	CCV-1
4	CCB-1
5	LCS
6	Method Blank
7	Sample
8	Sample Dup
9	Sample Spike
10	Sample
11	Sample
12	Sample
13	Sample
14	Sample
15	CCV-2
16	CCB-2
17-26	10 more Samples
27	CCV-3
28	CCB-3

Repeat steps 5-28 for remainder of samples.

TABLE 2

Summary of Corrective Actions

Method Reference	Control	Specification and Frequency	Acceptance Criteria	Corrective Action
353.2	ICAL	Prior to sample analysis	R ≥ 0.995	Correct problem then repeat ICAL
353.2	ICV	After ICAL	±10%	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
353.2	LCR	Initial and verified every 6 months	±10%	Re-establish
353.2	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL	If target exceeds MRL re-extract and re-analyze.
353.2	Matrix Spike	Include with each analysis batch (up to 10 samples)	Refer to ALS Kelso DQO Table	Evaluate data to determine if the there is a matrix effect or analytical error.
353.2	Sample Duplicates	Include with each analysis batch (up to 10 samples)	Refer to ALS Kelso DQO Table	Re-homogenize and re-analyze if result is > 5 X the MR.L
353.2	LCS	Include with each analysis batch (up to 20 samples)	Refer to ALS Kelso DQO Table	Re-extract and re-analyze.